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AIST ADVANCED TECHNOLOGY RESEARCH

JPRS-JST-90-013
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SCIENCE & TECHNOLOGY
JAPAN

AIST ADVANCED TECHNOLOGY RESEARCH

43067215 Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 1-477

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AIST Advanced Technology Research; Introduction

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Advanced Technology Themes

Symbol * in the remarks field refers to technical guidance

Classification of each technological field	Technological theme	Technical research institute name	Remarks
[Physics]			
1. Vibration, sound waves, supersonic waves, superflow frequency wave	•Research results on nonlinear waves	Mechanical Engineering Laboratory (Tsukuba) [MEL]	
	•Multi-dimensional normal-distribution irregular signal generator	Government Industrial Research Institute [GIRI], Kyushu	
[Metals]			
1. Powder metallurgy	•Plastic processing features of supereutectic Al-Si alloy	GIRI, Nagoya	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
	•Manufacturing shape-retaining alloy powder by rapid-cooling solidification	GIRI, Kyushu	
2. Manufacturing iron and iron alloys	•Composite materials based on heat-resistant alloys	GIRI, Nagoya	
3. Semiconductors	•Purifying semiconductors for use in optoelectronics and evaluative technique	Electrotechnical Laboratory (Tsukuba) [ET]	
[Metal Processing] 1. Casting			
	•Precision casting	GIRI, Nagoya	
2. Plastic processing	•Research on lubricating features of glass lubricant	MEL	*
	•Forming technique using superplasticity of titanium material	GIRI, Nagoya	
	•Extrusion processing of particle-scattered composite alloys	GIRI, Kyushu	
3. Welding and joining	•Joining different materials	GIRI, Shokoku	
4. Surface processing and plating	•Formation of corrosion- and abrasion-resistant titanium nitrate films by the plasma CVD method at low temperature	GIRI, Osaka	
	•Manufacturing technique of functional chromium thin films using the plasma CVD method	GIRI, Chugoku	
5. Corrosion and prevention of corrosion	•Hole corrosion prevention technique using ion penetrability of corrosion-resistant film	GIRI, Chugoku	
	•Corrosion damage evaluation of metal materials in high-speed flow	GIRI, Tohoku	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
5. [continued]	•Evaluation and preservation technique of hydrogen brittleness	GIRI, Chugoku	*
6. Other metal application techniques	•Underwater gas cutting technique for steel structure	GIRI, Shikoku	
	•Underwater arc dismantling technique for stainless steel	GIRI, Shikoku	
	•Manufacturing of permeable aluminum using liquid-phase sintered body	GIRI, Kyushu	
[Machines and Robots]			
1. Designing and manufacturing general machines	•Research on magnetic control of arc welding	MEL	
2. Machine parts and jigs	•Research on load capacity of small spur gear	MEL	
	•Developing oil-less ceramic bearing	GIRI, Osaka	
3. Robots	•Acquisition of techniques related to robot control	MEL	*
	•Developing intelligent sensor	Industrial Products Research Institute (Tsukuba) [IPRI]	
[Machining] 1. Precision machining	•Unified structure mechanism using elastic deformation as precision machine element	National Research Laboratory of Metrology [Tsukuba) [NRLM]	
	•Research on abrasion and lubrication in hot plastic processing	MEL	
2. Cutting and grinding	•Turning technique of cobalt-based alloy	GIRI, Chugoku	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
3. Special machining and laser machining	•Research on beam sound	MEL	
[Electricity, Communications, and Electronics]			
1. General power equipment	•Electrode technique in natrium thermoelectric power generation	Electrotechnical Laboratory	
2. Batteries and solar cells	•Lamination of temperature-difference cell	GIRI, Tohoku	
	•Power storing technique for solar cell by means of Redock's flow battery	EL	
	•New electrolyte plate for melted carbonate fuel battery	GIRI, Osaka	*
	•Accelerated life testing method for zinc-bromine battery	GIRI, Osaka	*
3. Optical communication and devices	•LB-film molecular orientation control technique	EL	
	•High-speed communication technique using optical space propagation	EL	
	•Hugh-performance magneto-optical material technique	EL	
4. Laser devices and optronic devices	•Research on light-reactive materials using molecular layout field	Chemical Engineering Laboratory [CEL]	
	•Research on hydrocarbon stability evaluation using optical reaction	National Research Institute for Pollution and Resources [NRIPR]	
5. Sensor and sensor application devices	•Magnetic sensor using superconductivity	GIRI, Chugoku	
[Measurement and Control]			
1. General measurement	•Measuring coefficient of linear expansion	NRLM	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
[1 continued]	•Automatic measuring of richly nutritious substances in lakes and marshes	GIRI, Osaka	
	•Underwater suspended particle measuring technique	GIRI, Chugoku	
	•Optical measuring of processed basic surface	GIRI, Chugoku	
	•Surface property measuring technique using electrons	GIRI, Chugoku	
	•Fuel density measuring technique using images	GIRI, Chugoku	
2. Measuring instruments	•Floor heating and snow-melting technique using heat pump—development of snow weight meter	Government Industrial Development Laboratory, Hokkaido [GIDL]	
	•STM with displacement measuring system	NRLM	
	•X-ray interferometer nanometer measurement	NRLM	
	•Development of practical use wavelength-stabilized laser	NRLM	*
	•Development of optical fiber composite sensor technology	EL	
	•Measurement and application techniques for Josephson effect	EL	
	•Development of β-ray absorbing continuous dust densitometer	NRIPR	*
	•Steel wire fatigue limit measuring system using personal computer	GIRI, Nagoya	
	•Precipitating particle collection device	GIRI, Chugoku	
	•Position detection technique using visual sensor	GIRI, Chugoku	
3. Process controller	•Product quality design system	NRLM	*
	•Measuring technique for grain size of suspended particles	NRLM	
	•High yield rate synthesizing process using superhigh pressure	CEL	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
[3 continued]	•Research on superhigh temperature generation, measurement, and use techniques	CEL	
	•Selectivity control of oxidizing reaction using composite oxide catalyzer	CEL	
[Mechatronics]			
1. Mechatronized devices	•Measuring technique for composite features of heat and moisture movement	GIRI, Osaka	
[Computers]			
1. Computer software	•Expert system technique for patent act	EL	
	•Software technique for uniformly mixing pottery stone	GIRI, Kyushu	
2. Image processing	•Simple robot driving technique using visual recognition processing	GIRI, Shikoku	
[Information Processing]			
1. Information processing system	•Development of deep-sea towing seismic wave proving device	Geological Survey of Japan (Tsukuba) [GSJ]	
2. Information processing technology	•Syntax of natural language and meaning analysis technique	EL	
[Basic Chemistry]			
1. Radiation chemistry	•Classification of reaction mechanism by using regulated field	GIRI, Nagoya	
2. General analytical chemistry	•Quantitative analysis of titanium nitride thin film	GIDL	
	•Research on element analysis of very small-quantity dust	GIDL	
	•Analysis and measurement of physical and chemical properties of filter paper	GIDL	*

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
[2 continued]	•Quantitative analysis of chlorine in silicon nitride	GIRI, Nagoya	
	•Technique to analyze light absorption of atoms in carbon furnace	GIRI, Chugoku	*
	•Method of determining complex radical formation stability by potential titration	GIRI, Shikoku	
3. Macro-molecule chemistry	•Method of manufacturing new (bi) chromic acid in absorbent	GIRI, Tohoku	
	•Composite absorbent based on acrylic acid ester	GIRI, Tohoku	
4. Physical properties of macro-molecule	•New development in polymer molecular weight measurement using liquid chromatography	GIRI, Nagoya	
5. Macro-molecular reaction	•New substance synthesis using solid phase polymerization reaction under superhigh pressure	CEL	
6. Polymerization reaction	•Techniques for synthesis and polymerization of acetylenes having fluorine-containing hetero-ring	GIRI, Nagoya	
[Chemical Engineering]			
1. General chemical engineering	•Opening new reaction group by activation of C-H connection	CEL	
2. Chemical engineering materials	•Composite accumulated film creation device	Research Institute for Polymers and Textiles [RIPT] (Tsukuba)	
3. Heat exchanger and cooler	•Research on heat pipe for high temperatures	NRIPR	*

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
4. Combustion apparatus	•Flowing combustion of powdered coal	GIDL, Hokkaido	
	•Research on combustion technique for wood fuel	NRIPR	
5. Solid processing device	•Technique for synthesis of superconductive thin film	CEL	*
	•Research on carbon materials—manufacturing of hard carbon film by plasma CVD and its physical properties	NRIPR	*
6. Powder engineering and powder device	•Basic research on fine grain manufacturing technique using plasma method	NRIPR	
	•Opening meshes of plane-mat woven net	GIRI, Nagoya	
	•Dry separation system for coal ash	GIRI, Kyushu	
7. Solid processing device	•Crushing GFRP with rotary shearing crusher	GIRI, Shikoku	
[Chemical Industry]			
1. Rubber and plastics industry	•Research on advanced composite materials	MEL	*
	•Research on synthesis and composition of macro-molecules by RIM	GIRI, Osaka	
	•Measuring the degree of orientation of plastic and composite materials by minute shearing method	GIRI, Osaka	
	•Structure of reinforcement material using fiber reinforcement resin and composing technique	GIRI, Osaka	*
2. Manufacturing process and equipment	•Influence of spinning conditions on layout of pitch-based carbon fibers on carbon layer surface	GIRI, Kyushu	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
3. Application products and half-finished products	•Implementation of naphthalene and camphor by assisting agent	GIRI, Osaka	
	•Iodine-adsorbing behavior of carbon mesophase	GIRI, Kyushu	
4. Ceramic industry	•Carbon having boron as solid solution	GIRI, Kyushu	
5. Glass and enamel	•Alkali-resistant porous glass	GIRI, Osaka	
	•Technique to develop glass material under zero gravity conditions	GIRI, Osaka	
	•Sound wave floating melting technique	GIRI, Osaka	*
6. Ceramics	•Synthesis of steatite porcelain using talc produced in Matsumae	GIDL	
	•Technique to synthesize artificial clay	GIRI, Nagoya	*
7. Lime and cement	•Extruded building material based on cement reinforced by composite synthesized fiber	GIRI, Kyushu	*
8. New ceramics	•Synthesis of $SiC-Si^2N^4$ composite fine-grain powder	GIDL	
	•Creation of functionally gradient materials by self-propagating high temperature synthesis	GIRI, Tohoku	
	•Ceramics coating technique for inside of bent pipe by reaction melting and adhesion	GIRI, Tohoku	*
	•Diffusion joining of ceramics	GIRI, Nagoya	
	•Dynamic fatigue test of ceramics at room temperature	GIRI, Nagoya	
	•Ejection forming of ceramics and metal powder	GIRI, Nagoya	
	•Manufacturing of high-temperature oxide superconductive material and its electromagnetic properties	GIRI, Nagoya	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
[8 continued]	•Clay layer compounds with mesospores and their manufacturing methods	GIRI, Nagoya	
	•Creation of Y-Ba-Cu-O-based superconductive thin film by ion beam spattering method and evaluation of the film	GIRI, Nagoya	
	•Synthesis of needle-like Al_2O_3 fine particles	GIRI, Nagoya	
	•Oxidation reaction and EPMA observation of silicon nitride	GIRI, Nagoya	*
	•Manufacturing of oxide optical semiconductor	GIRI, Nagoya	*
	•Establishment of mud loading method for highly pure alumina	GIRI, Nagoya	*
	•Synthesis conditions for calcium phosphate compound	GIRI, Nagoya	*
	•Method for joining single crystal MgO	GIRI, Osaka	*
	•Increase in ion conductivity of solid electrolyte pellet by spread of dielectrics	GIRI, Osaka	
9. Organic chemistry and fuel engineering	•Synthesis of new (pelfluro-bicyclolactam)	GIRI, Nagoya	
	•Analysis of organic reaction by chemical ionization mass analysis method		
[Bionics]			
1. General biochemistry	•Adjusting factor of (hialron) acid synthesis (HASI)	Fermentation Research Institute [FRI] (Tsukuba)	
2. Biochemistry of microbes	•Long preservation of frozen, dried, and sensitive germ	FRI	
	•Clarification of damage mechanism of microbe cell during frozen preservation	FRI	
	•Corpulent cell degranulation suppressing materials produced by microbes	FRI	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
[2 continued]	•Material conversion from hydrocarbons by microbes	FRI	
	•Decomposition of lignin model material by aragekawara mushroom	GIRI, Shikoku	
3. Animal biochemistry	•Building up skin cells in blood vessels and production of shrinkage peptide	FRI	
4. Genetic engineering	•Ionization and ion decomposition of samples related to living bodies	CEL	
	•Substance production by metabolic genes	FRI	
	•Gene cloning by highly effective shape/quality transformation system involving highly thermophilic Thermus bacteria	FRI	
5. General bioengineering and biomass	•Selective extraction and separation of EPA.DHA from fish testes with supercritical carbonic acid	GIRI, Tohoku	
	•Research on conversion of plant biomass to carbohydrate with microbes	FRI	
	•Countercurrent oils and fats continuous decomposition bioreactor using solidified lipase	FRI	
	•Laver mass cultivation device	GIRI, Chugoku	
	•Technique for fixing bacteriophage	GIRI, Chugoku	*
6. Fermentation industry	•Technique for secretion and production of different protein with yeast	CEL	*
	•Production of heat-resistant lipase and decomposition of fats with yeast	FRI	*
[Forestry and Fisheries]			
1. Fisheries engineering	Trial manufacturing and study of fiberform medium	GIRI, Kyushu	*

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
[Medical Science and Pharmacy]			
1. General medical science and pharmacy	•Synthesis and properties of pyrimidine platina origomar complex radical	CEL	
2. Medical devices and instruments	•Research on design and processing technique of advanced medical composite materials	MEL	
3. Medical and pharmaceutical system devices	•New use technology of silk protein	IPRI	*
[Wood and Paper Manufacture]			
1. Wood chemistry and products	•Manufacture of activated charcoal with lumber fron thinning as raw material	GIDL	*
2. Pulp industry	•Manufacturing technique for yeast pulp paper (breaching)	GIRI, Shokoku	
[Fiber]			
1. Physical properties of fiber	•Fixing of living body catalyst with polyvinyl alcohol extrafine fiber (SFF)	RIPT	
2. Fiber product apparel industry	•CAD system for apparel	RIPT	
[Construction]			
1. Civil engineering materials	•Durability evaluation technique for GRC	IPRI	
[Environmental, Pollution, Satellite and Radiation]			
1. General environment engineering	•Method of removing poisonous chlorine compound using supercritical carbonic acid gas	GIRI, Tohoku	
	•Control technique for in-gulf flow state by hydraulic model experiment	GIRI, Chugoku	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
2. Waste water processing	•Waste water processing technique for small-scale fisheries processing factory in cold-weather district	GIDL	
	•Waste water processing technique for medium-scale fisheries processing factory in cold-weather district	GIDL	
	•Method of removing fluorine ions in lanthanum (III)—carrying chelate resin	GIRI, Tohoku	
	•High-level processing of nitrogen by method involving adding drained water separately	FRI	
	•Processing technique for non-electrolytic-plating method	IPRI	
	•Research on functional resin for water processing	NRIPR	
	•Research on high-level processing of difficult-to-separate organic compounds	NRIPR	
	•High-gradient magnetic sorting of mineral products	NRIPR	
	•Drained water processing technique using anaerobic fermentation	GIRI, Osaka	
	•Processing technique for canned-orange waste water	GIRI, Shikoku	
	•Activated carbon adsorption processing or drained water containing chromatic acid ions	GIRI, Kyushu	
3. Disposal of waste and dust	•Research in processing of industrial waste containing organic chlorine	NRIPR	
4. Water pollution	•Removing minute particles by magnetite seed method	GIRI, Tohoku	
	•Ion chromatography of water pollution components	GIRI, Nagoya	
5. Unpleasant odors	•Method to remove unpleasant odors by employing microbes	FRI	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
6. Environment measuring device system	•Bottom water layer oxygen consumption speed measuring device	GIRI, Chugoku	
7. Environment assessment	•Analysis of corrupt substance spread pattern	GIRI, Chugoku	
[Energy and Resources]			
1. General resources	•Lithium adsorbent effective at high temperatures		
	•Manufacturing technique for new metal ion adsorbent	GIRI, Tohoku	
	•Technique for separating gadolinium and yttrium		
	•Development of virtual base displacement method system	Geological Survey of Japan [GSJ], Tsukuba	
	•Ore dressing of rare earth minerals and solvent extraction of nonferrous metals		
	•Research on rare metal refining technique	NRIPR	*
	•Research on effective use of clay minerals		
	•Lithium recovery technique using titanic acid fibers	GIRI, Shikoku	
	•Technique for recovery of boron from the water accompanying natural gas	GIRI, Kyushu	
2. Fossil fuel energy	•Floating combustion technique for peat—dehydrating peat	GIDL	
	•Separation and characterization of polar compound in artificial naphtha by adsorption method	GIDL	
	•Research on lightening heavy oil	NRIPR	
	•Research on technique for making inferior oil nonpollutive	NRIPR	
	•Research on mixing coal and solvent with ESR	GIRI, Kyushu	

[continued]

[Continuation of Advanced Technology Themes]

Classification of each technological field	Technological theme	Technical research institute name	Remarks
3. General energy saving	•System simulation for heat pump in cold-weather districts	GIDL	
4. Reuse of wastes	•Technique for converting fisheries processing waste into livestock feed by solid fermentation method	GIDL	
	•Technique for processing and using redundant mud by low-temperature methane fermentation method	GIDL	
	•Technique for converting plastic waste to light oil	GIDL	*
	•Flowing combustion technique for chaff	GIDL	*
	•Microporous material synthesis	CEL	
	•Research on technique for solidifying and using coal ash effectively	CEL	
	•Research on processing heavy oil and substitute fuel for petroleum	NRIPR	
	•Research on sugar-accumulation active sludge method	NRIPR	
	•Research on technique for conversion of coke furnace by-product tar fraction to new resources	NRIPR	
	•Research on filtration assistant using fly ash	GIRI, Kyushu	
[Product Planning and Distribution]			
1. Convergence and transportation	•Research on measurement and control of vehicles	MEL	
	•Research on fuel consumption testing method for automobiles	MEL	
[Production]			
1. Materials	•Superconductive thin film material technique	EL	
[Production Control, Management and Safety]			
1. General production control	•Research on work analysis for automation	MEL	

When Using This Advanced Technology Application Handbook (1986 Version)

This handbook classifies and summarizes, for each technical field, the technical themes which are thought of as having widespread effects and are usable for and applicable to small and medium enterprises, from among the advanced technologies-related research results, expertise, and technical guidance cases which are being held by local experimental institutes (local institutes) and The 9th Testing and Research Institute, Tsukuba (Tsukuba Institute) under the Agency of Industrial Science and Technology.

Table 1 classifies these technical themes for each technical field and each institute.

For more detailed information on any of the technical themes covered in this handbook, please contact the Technical Interchange Promotion Center for the local institutes or the Technical Clinic for Tsukuba Institute.

Table 1.1 Classification of Advanced Technology Research Results for Each Technical Field and Institute

	Overall		AIST, District test and research institute name													
	Overall		GIDL, Hokkaido		GIRI, Tohoku		GIRI, Nagoya		GIRI, Osaka		GIRI, Chugoku		GIRI, Shikoku		GIRI, Kyushu	
	No of research results	No of technical guidance cases	No of research results	No of technical guidance cases	No of research results	No of technical guidance cases	No of research results	No of technical guidance cases	No of research results	No of technical guidance cases	No of research results	No of technical guidance cases	No of research results	No of technical guidance cases	No of research results	No of technical guidance cases
Physics	1														1	
Metals	3						2								1	
Metal processing	11	1			1		2		1		2	1	3		2	
Machines and robots	1								1							
Machining	1										1					
Electricity, communications, electronics	2	2			1					2	1					
Measurement and control	9		1				1		1		6					
Mechatronics	1								1							
Computer	2												1		1	
Basic chemistry	8	1	1		2		4					1	1			
Chemical engineering	4		1				1						1		1	
Chemical industry	21	10	2		1	1	9	5	6	3					3	1
Bionics	3	1			1						1	1	1			
Forestry and fisheries		1														1
Wood and paper manufacturing	1	1		1									1			
Environment, pollution, sanitation & radiation	11	1	2		3		1			1	3		1		1	
Energy and resources	12	2	5	2	3								1		3	
Total	91	20	12	3	12	1	20	5	10	6	14	3	10	0	13	2

Table 1.2 Classification of Advanced Technology Research Results by Technical Field and Institute

			AIST, The 9th Testing and Research Institute, Tsukuba																	
	Overall		Electro-technical Laboratory		Mechanical Engineering Laboratory		Industrial Products Research Institute		National Research Institute of Metrology		Geological Survey of Japan		National Research Institute for Pollution and Resources		Chemical Engineering Laboratory		Research Institute for Polymers & Textiles		Fermentation Research Institute	
	Research results	Technical guidance cases	Research results	Technical guidance cases	Research results	Technical guidance cases	Research results	Technical guidance cases	Research results	Technical guidance cases	Research results	Technical guidance cases	Research results	Technical guidance cases	Research results	Technical guidance cases	Research results	Technical guidance cases	Research results	Technical guidance cases
Physics	1				1															
Metals	1		1																	
Metal processing		1				1														
Machines and robots	3	1			2	1	1													
Machining	2	1			2					1										
Electricity, communications, electronics	7		5										1		1					
Measurement and control	9	3	2						4	2				1	3					
Computer	1		1																	
Information processing	2		1								1									
Basic chemistry	2	1											1	1	1					
Chemical engineering	4	3											2	2	1	1	1			
Chemical industry		1				1														
Bionics	11	2													1	1			10	1
Medical science and pharmaceuticals	2	1			1			1							1					
Fibers	2																2			
Construction	1						1													
Environment, pollution, sanitation and radiation	7						1						4						2	
Energy and resources	10	1									1		7	1	2					
Product planning and distribution	2				2															
Production	1		1																	
Production control, management and safety	1				1															
Total	69	15	11	0	9	3	3	1	4	3	2	0	15	5	10	2	3	0	12	1

Table 2. Locations and Contacts of the Testing and Research Institutes Belonging to the Agency of Industrial Science and Technology, Objects of Investigation While Preparing This Handbook

Test and research institute	Address	Phone	Contact
Government Industrial Development Laboratory, Hokkaido	061-01 2-17-2-1 Tsukisappu-higashi Toyoira-ku Sapporo-shi	011(851)0151	Technology Interchange Promotion Center
Government Industrial Research Institute [GIRI], Tohoku	983 4-2-1 Koketake Hara-machi Sendai-shi	0222(37)5211	" " "
GIRI, Nagoya	462 1-1 Hirate-cho Kita-ku Nagoya-shi	052(911)2111	" " "
GIRI, Osaka	563 1-8-31 Midorigaoka Ikeda-shi	0727(51)8351	" " "
GIRI, Chugoku	737-01 15,000 Hiro-machi Kure-shi	0823(72)1111	" " "
GIRI, Shikoku	760 2-3-3 Hananomiya-cho Takamatsu-shi	0878(67)3511	" " "
GIRI, Kyushu	841 Yado-machi Torisu-shi	0942(82)51615	" " "
National Research Laboratory of Metrology	305 1-1-4 Umezono Tsukuba-shi	0298(45)4117	Measuring Eng. Clinic
Mechanical Engineering Laboratory	305 1-2 Namiki " "	0298(54)2524	Mechanical Eng. Clinic
Chemical Engineering Laboratory	305 1-1 Higashi " "	0298(54)4423	Chemical Eng. Clinic
Fermentation Research Institute	305 1-1-3 " " "	0298(54)6024	Technical Clinic
Research Institute for Polymers and Textiles	305 1-1-4 " " "	0298(54)6245	
Geological Survey of Japan	305 1-1-3 " " "	0298(54)3540	Geological Clinic
Electrotechnical Laboratory	305 1-1-4 " " "	0298(54)5014	Technical Clinic
Industrial Products Research Institute	305 1-1-4 " " "	0298(54)6607	Product Chem.Clinic
National Research Institute for Pollution and Resources	305 16-3 Onogawa " "	0298(54)3036	Technical Clinic

Dissemination of Research Results, Outside Contacts by AIST

43067215b Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 5-7

[Text] The Agency of Industrial Science and Technology has nine government industrial research institutes (institutes) in the Tsukuba Research Center and seven local institutes, representing Japan's national testing and research institutes related to the mining and manufacturing industries, and is promoting research on: 1) important political subjects related to energy, resources and environmental integrity; and 2) basic national concerns, such as measurement standards and geological maps. Since advanced technology development is thought to be especially important for Japan in its attempt to establish a technological state, this agency is also positively grappling with the development.

The district testing and research institute (district institute) which has been established for each district, is not only grappling with national-level technological subjects as a district office of a national institute, but is also promoting the R&D of district concerns, such as the enhancement of local industries and the effective use of local resources to contribute to the development of the district.

Efforts have been made to spread and transfer these results extensively via research results presentations, issuing reports of research results, or technical consultation and guidance.

The following system is also available as a means of cooperation and support for private industries' technological development in addition to the above-mentioned spread and transfer of results.

1. Cooperative Research

Research activities for new technology development require efficient and concentrated investment in the research staff and research projects and consistent balanced development from basic research to industrialization. As a part of the measure for this requirement, the Agency of Industrial Science and Industry is operating a cooperative research system involving an institute and a private enterprise.

Cooperative research is first thought necessary in settling the problems involving the boundary region between application and industrialization research. The fields where cooperative research is effective are:

(1) Those requiring substantial funds or much labor, such as industrialization tests with test plants or tests utilizing cooperative test equipment,

(2) Those requiring the technical cooperation of many related departments, or those which need the cooperation of many technical experts, such as special and high-level techniques,

(3) Common research subjects that extend over multiple industrial fields and subjects requiring extremely basic research.

These types of research are thought of as being difficult for an enterprise to perform alone.

From the above-mentioned viewpoint, institutes have been engaging in cooperative research with universities and private enterprises since 1957 and will strongly promote this system in the future.

Cases Involving Cooperative Research During Past 3 Years

FY	Total	District institute
1985	65 cases	33 cases
1986	71 cases	40 cases
1987	76 cases	31 cases

Incidentally, when a cooperative research plan is put forward, a cooperative research application is to be submitted and a cooperative research contract to be exchanged.

2. Entrusted Research

The institute is performing research in the range accepted as being part of the duty of a national research institute, and these research subjects are basically common in that they are being used by many unspecified individuals and organizations. From the standpoint of related enterprises, these research subjects can, in many cases, be transformed into those necessary for the enterprises themselves if specific conditions are met or the range of the setup conditions is limited. Also, an enterprise which encounters a problem but cannot afford its own research organization to solve the problem cannot but entrust the research to the outside.

Table of Technical Guidance and Consultation During Past 3 Years

(Number of cases)

Testing and research institute name / FY	1985		1986		1987	
	Technical guidance	Consulting	Technical guidance	Consulting	Technical guidance	Consulting
National Research Laboratory for Metrology	28	964	23	1,254	23	1,400
Mechanical Engineering Lab	98	509	100	736	95	672
Chemical Engineering Lab	139	602	143	1,499	143	1,184
GIRI, Osaka	175	3,437	167	3,421	154	2,744
GIRI, Nagoya	165	1,440	145	1,587	124	1,778
Fermentation Research Institute	42	1,054	60	1,128	55	1,331
Research Institute for Polymers and Textiles	52	340	45	374	59	335
Geological Survey of Japan	71	1,265	15	1,535	75	1,734
Electrotechnical Laboratory	156	600	134	650	126	660
Industrial Products Research Institute	41	387	41	375	29	450
National Research Institute for Pollution and Resources	47	460	42	408	41	357
Government Industrial Development Laboratory, Hokkaido	28	311	44	323	38	338
GIRI, Kyushu	36	387	28	908	28	738
GIRI, Shikoku	26	453	28	472	15	460
GIRI, Tohoku	11	174	19	134	16	122
GIRI, Chugoku	15	208	18	183	17	158
Total	1,130	12,591	1,052	14,987	1,038	14,461

In other words, if the research topics of the institutes belonging to the Agency of Industrial Science and Technology are announced, enterprises can obtain business guidelines or have a sense of being economical or of well being when confronting some bottlenecks, but will be unable to resolve them.

In 1957, the Agency of Industrial Science and Technology itself established a system allowing the agency to receive trust from private enterprises and to conduct research when useful data could be offered directly to the enterprises

by slightly extending current research contents or by making some modifications to the existing equipment.

This system is called entrusted research, and when research is initiated, an entrusted research contract will be exchanged to determine the research contents, research institute, expenses for the research, and the handling of cases in which patent is involved.

Technical Guidance

The institute conducts basic and common research, and one of the duties assigned to the agency is to make the long accumulated techniques and expertise, i.e., research fruits, extensively available.

On the other hand, related enterprises are strongly requesting technical consultation and guidance from the institutes conducting research involving basic and common foundations.

In 1957, therefore, the agency established and internal industrial technology clinic and a technical clinic in each institute to practice technical guidance and consultation. In the district institutes, the technical clinics were renamed "technical interchange promotion centers" on 1 July 1986 for expansion and reorganization purposes.

4. Requested Tests and Analyses and Use of Equipment

The institute performs tests (including type tests) and analyses and issues judgments when requested by private enterprises, and issues certificates. Also, if an enterprise issues a request to use an institute's equipment, the institute will lend the equipment or allow the enterprise to use it as long as it does not disrupt the institute's research.

List of Entrusted Testing and Analysis Jobs for Testing and Research Institutes of the Agency

National Research Institute for Metrology	3,157
Mechanical Engineering Laboratory	14
Chemical Engineering Laboratory	30
Government Industrial Research Institute, Osaka	16
Government Industrial Research Institute, Nagoya	154
Fermentation Research Institute	36
Research Institute for Polymers and Textiles	20
Geological Survey of Japan	0
Electrotechnical Laboratory	178
National Institute for Pollution and Resources	377
Industrial Products Research Institute	19
Government Industrial Development Laboratory	115
Government Industrial Research Institute, Kyushu	46
Government Industrial Research Institute, Shikoku	0
Government Industrial Research Institute, Tohoku	16
Government Industrial Research Institute, Chugoku	4

Incidentally, appropriate fees are collected for the above labor.

5. Industrial Property

For an invention, idea, or creation realized by an institute staff member, the director of the Agency of Industrial Science and Technology approves the right to receive a patent on the basis of the Agency of Industrial Science and Technology's duty invention regulation and submits applications domestically and overseas.

The application and acquisition statuses of industrial properties as of 31 March 1988 are shown in the following tables.

Many of the industrial properties owned by the agency are already being used effectively by private enterprises, with more expected to be used increasingly extensively.

Industrial Property

Type	Owned	Applied for
Foreign patents	1,069	634
Domestic patents	6,628	7,679
Utility models	443	442
Design rights	58	6
Total	8,198	8,761

Status of Practice (Practice domestically and abroad)

Patent		Utility model		Design right		Total	
Number of rights	Practicing companies (Total)	Number of rights	Practicing companies (Total)	Number of rights	Practicing companies (Total)	Number of rights	Practicing companies (Total)
697(10)	923(10)	49	64	2	4	748(10)	991(10)

Manufacturing Shape Memory Alloy Powder by Rapid-Cooling Solidification Method

43067215c Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 19-21

[Article by Government Industrial Research Institute, Kyushu]

[Text] **Summary**

The authors manufactured shape memory alloy powders, kinds of functional materials, by the rapid-cooling solidification method. Shape-memory alloys based on Ni-Ti alloys and on copper are currently on the market, but copper-based shape-memory alloys are inferior to those based on Ni-Ti alloys in that their fatigue lives are shorter. However, their cost is as low as about one-tenth that of shape-memory alloys based on Ni-Ti, demonstrating the possibility of lowering their prices. Since copper-based alloys have remarkably high thermal and electric conductivities, they are suitable for electronic parts and allow manufacturers to obtain devices with superior responsiveness.

The shorter fatigue lives, weak points of copper-based shape-memory alloys, can be improved to some degree by making the metal structure minute. In this research, therefore, the authors manufactured copper-based shape-memory alloy powder by the rapid-cooling solidification powder method (rotary-disk atomize method), which is a very effective means of minimizing the size of metal structures, in an attempt to make the metal structures minute.

Detailed Contents

Recently, great expectations have been placed on new metallic materials to play important roles for various purposes. New metallic materials are roughly divided into structural and functional materials. The materials that have features far superior to those of the materials which have been used so far, such as alloys of high specific strength and a high modulus of elasticity, super-heat-resistant alloys, and alloys for use at extremely low temperatures, for example, are classified as structural materials. On the other hand, materials that have not existed before and have totally new functions and properties, such as superconductive alloys, amorphous alloys, superplastic alloys, shape-memory alloys, and alloys that absorb and store hydrogen, are classified as functional materials. This research pays attention to shape-

Table 1. Examples of Shape Memory Alloys

Alloy system	Composition	Af_s°C	A_s°C
Ti-Ni	Ti-50 Ni at%	60	78
	Ti-51 Ni at%	-30	-12
Ti-Ni-Cu	Ti-20Ni-30 Cu at%	80	85
Ti-Ni-Fe	Ti-47 Ni-3 Fe at %	-90	-72
Cu-Zn	Cu-39.8 Zn wt%	-120	—
Cu-Zn-Al	Cu-27.5 Zn-4.5 Al wt%	-105	—
	Cu-13.5 Zn-8 Al wt%	146	—
Cu-Al-Ni	Cu-14.5 Al-4.4 Ni wt%	-140	-109
	Cu-14.1 Al-4.2 Ni wt%	2.5	20
Cu-Au-Zn	Au-21 Cu-49 Zn at%	-153	—
	Au-29 Cu-45 Zn at%	57	—
Cu-Sn	Cu-15.3 Sn at%	-41	—
Ni-Al	Ni-36.6 Al at%	60±5	—
Ag-Cd	Ag-45.0 Cd at%	-74	-80
Au-Cd	Au-47.5 Cd at%	58	74
In-Tl	In-21 Tl at%	60	65
In-Cd	In-4.4 Cd at%	40	50

memory alloys from among functional materials, and describes examples of manufacturing the alloys by the rapid-cooling solidification method.

The shape-memory alloy is a material which, if a specific shape is given in advance, remembers the shape and returns to the remembered original shape when heated, even if it has been deformed. As materials which show such unique move, the alloys shown in Table 1 as examples, have been developed so far. Of these alloys, only two are currently on the market: the Ni-Ti alloy and the Cu-Zn-Al-based shape-memory alloy, of which the Ni-Ti alloy has produced many results through actual use. This is because it has such features as:

(1) high shape-recovered ability (maximum deformation 7~8 percent),
(2) large recovery stress (up to about 60 kg/mm^2),
(3) long fatigue life (10^5 times at 2 percent deformation),
(4) superior abrasion resistance and corrosion resistance which is friendly to the human body.

On the other hand, the copper-based shape-memory alloy readily causes destruction from the grain boundary of the crystal, and the Cu-Al-Ni based alloy has a particular weak point in that its fatigue life is short. The Cu-Al-Ni-based alloy has another weak point in that its machinability at high and low temperatures is bad compared with that of the Cu-Zn-Al alloy. However, since the copper-based alloy is priced at as low as about one-tenth that of the Ni-Ti alloy, if the above-mentioned weak point is improved to some degree, there is the possibility that it will be commercialized in the fields n which the manufacturers are currently to put the Ni-Ti alloy to practical use due to its high cost. Also, the copper-based alloy has far higher thermal and electric conductivities than those of the Ni-Ti alloy and, because of this,

allows devices superior in responsiveness to be manufactured. Therefore, if the problems with the above-mentioned fatigue life and machinability in the Cu-Al-Ni based alloy are resolved, the fields of practical use will be expanded further.

It is being made clear by the research completed so far that the fatigue life of the copper-based shape-memory alloy can be improved by making the metal structure minute. Furthermore, a minute metal structure also improves machinability. The rapid-cooling solidification method, which is adopted in this research, is a very effective technique for making metal structures minute. If powder is used as the starting raw material, near-net-shape machining becomes possible, and from this point also, the machinability problem in the Cu-Al-Ni-based alloy is reduced.

A method of manufacturing powder by blowing air or water onto the melted alloy, then atomizing and solidifying it, has been used in the past and is called the atomizing method. If this method is used, the volume of powder is very small compared with the ingot in the melting casting method. Therefore, the powder is cooled rapidly. The rapid-cooling solidification method promotes this concept and makes the cooling speed during powder manufacturing remarkably faster.

If the rapid-cooling solidification powder method is used, segregation does not occur in the powder metal structure, and metal structures which are very minute and uniform and solid-solution structures supersaturated with alloy elements are obtained. If the rapid-cooling solidification powder method producing such a superior structure is improved so as to be able to yield a gapless and compact formed body while, at the same time, attaining a metal structure without having degraded it, a metal structure superior in such mechanical properties as strength, toughness, thermal resistance and abrasion resistance will be obtained. In some cases, such capabilities as superplasticity will be displayed.

Various methods have been proposed and studied to obtain rapid-cooling solidification powder. These methods mostly use "rotation," as exemplified by the rotary disk atomizing method, rotary electrode method, rotary crucible method, rotary cup method, and roller atomizing method. Our institute adopted the rotary disk atomizing method since it is capable of the most effective powder manufacturing methods, and is developing various rapid-cooling solidification powders. This article describes only the manufacturing conditions of copper-based shape-memory alloy powder and the metal structure of the obtained powder.

To manufacture copper-based shape-memory alloy powder by the rotary disk atomizing method (Figure 1), the inside of the chamber is first made a vacuum or an inert gas atmosphere, then the ground metal in the crucible is melted. The authors are using a high-frequency induction heating method in consideration of the cleanliness of the chamber and the output. The melted copper-based alloy is jetted onto a disk, which is rotating at a high speed (20,000 rpm or more), and is atomized by the centrifugal force of the rotating disk. The atomized alloy is rapidly cooled and solidified, while splashing

inside the chamber, by the cooling gas which flows from the primary to tertiary nozzles which have been circumferentially set, and clean-surface copper-based alloy powder is obtained. Table 2 shows sample manufacturing conditions of the powder.

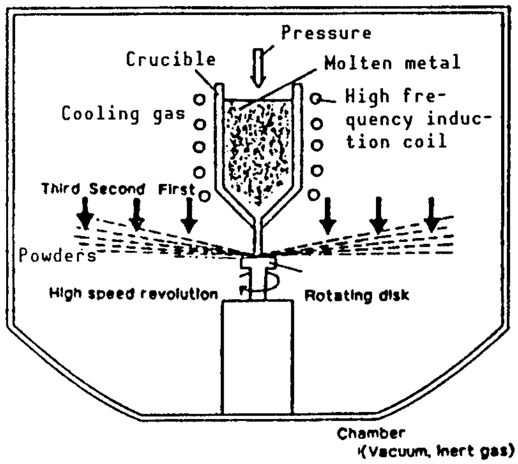

Figure 1. Rotating Disk Atomizing Method

Table 2. Example of Manufacturing Conditions for Copper-Based Shape-Memory Alloy Powder

Item		Manufacturing condition
Melting	Melting quantity	600 gf
	Melting temperature	1,230°C
	Holding time	6 min
	Melting atmosphere	He gas (in melting crucible or chamber)
Ejection	Nozzle diameter	3 mmϕ
	Ejection system	Stopper removal method
Atomizing/ rapid-cooling solidification	Disk shape	Flat type
	Disk material	ZrO_2
	Number of rotations of disk	22,000 rpm
	Cooling gas	He gas

Photograph 1 [not reproduced] shows an example of manufacturing Cu-13Al-4Ni shape-memory alloy powder using the rotary disk atomizing method, while Photograph 2 [not reproduced] shows that of manufacturing Cu-21Zn-6Al shape-memory alloy powder. These photographs also show the metal structures of these melted casting materials for comparison and reference. In both

Photographs 1 and 2 [not reproduced], (a) shows the external appearance of the rapid-cooling solidification powder, which is almost spherical, (b) shows the metal structure of the rapid-cooling solidification powder, with both photographs indicating that this powder is of a very minute metal structure, while the metal structure of the melted casting material shown in (c) is coarse due to the slow cooling speed.

In this way, it has been proved that if the rotary disk atomizing method which has been adopted in this institute is used, a powder with a superior metal structure is obtained. The authors are scheduled to continue their detailed examination of the forming processing and the shape-memory characteristic of the copper-based shape-memory alloy powder. The authors also intend to examine not only the copper-based shape-memory alloy powder, but also the Ni-Ti based and iron-based shape-memory alloys.

Table 3[1] shows sample applications of shape-memory alloys which have already been proposed or put to practical use. The application fields of shape-memory alloys are expected to spread further with the further improvement of the shape-memory characteristic and mechanical properties.

Table 3. Application Examples of Shape-Memory Alloy as Classified by Use

	One direction	Two directions
Without load	Antenna for spaceship Condensation filter Lead time for integrated circuit	Automobile carburetor
Load	Connector Coupling Clamp	Various thermally-driven elements Greenhouse window switch Damper for air conditioner Radiator valve Fan clutch for automobile Safety switch Recorder pen drive unit Heat engine

References

1. NIKKEI NEW MATERIALS, 24 February 1986, p 82.

Composite Materials Based on Heat-Resistant Alloys

43067215d Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 22–23

[Article by Government Industrial Research Institute, Nagoya]

[Text] **Summary**

As a method of further enhancing the toughness of heat-resistant alloys in a high-temperature range, research is being promoted on composing fibers of various heat-resistant metals and ceramics in an alloy whose matrix is Fe, Co, Ni or Ti.

In this research, the authors examined the boundary-surface reaction and difference in thermal expansion coefficient which represent problems when fibers are composed in a heat-resistant alloy, and especially, investigated the W fiber, which is thought promising for use in the movable blade of the gas turbine engine in aircraft.

The W fiber becomes stronger as it becomes thinner, but usually, it is of 0.1 mmϕ and doped with very small quantities of Al, K and Si to raise the secondary crystallization temperature, and contains a small quantity of ThO_2 or Re to form a solid solution. However, since W is of large specific gravity, it can be used effectively in combination with Mo metal fiber or ceramic fibers of C, SiC, B, or Al_2O_3, whose specific gravity is comparatively small.

Detailed Contents

To enhance the thermal efficiency and durability of various internal combustion engines, it is important to develop materials that can endure higher temperatures. Aircraft gas turbine engine parts in particular are used under severe conditions and must endure creep at high temperatures and high pressure and the thermal fatigue caused by heating and cooling. Therefore, the development of materials superior in thermal resistance is expected.

The method conceived to enhance the toughness of heat-resistant alloys at high temperatures, involves generating metals and ceramic fibers, as shown in Table 1, whose melting points are high. Table 2 shows sample combinations of various fibers and matrices which are being studied as composite materials.

When a fiber suitable for a matrix is selected, both need to be examined for boundary-surface reactions at high temperatures. For example, if Ni is contained in a matrix, the Ni is known to diffuse and penetrate into the W fibers at high temperatures and destroy the fibered, processed structure, a characteristic of the fiber. To prevent this undesirable phenomenon, some people are studying the method of coating in thermodynamically stable substance, for example, oxide, carbide, or nitride of Zr, Hf, or Ti, on the surface of the W fiber in advance by the CVD or PVD method.

Table 1. Characteristics of High-Melting Point Metal Ceramics

Metal	Melting point (°C)	Specific gravity	Ceramics	Melting point (°C)	Specific gravity
W	3,380	19.3	C	3,700	2.2
Re	3,180	21.0	ToC	3,140	4.2
Os	3,045	22.5	ThO_2	3,050	10.0
Ta	2,990	16.6	BN	3,000*	2.1
Mo	2,630	10.2	MgO	2,800	3.6
Nb	2,520	7.0	SiC	2,700	3.2
Ir	2,443	22.4	CaO	2,570	3.3
Ru	2,250	12.2	BeO	2,570	3.0
Hf	2,230	13.3	ZrO_2	2,550	5.6
Tc	2,170	11.5	AlN	2,500*	3.1
Rh	1,960	12.4	B_4C	2,450	2.5
Cr	1,890	7.2	ZrO_2 SiO_2	2,420	4.7
Zr	1,855	6.5	Y_2O_3	2,410	4.5
V	1,835	6.1	B	2,064	2.5
Pt	1,769	21.5	Al_2O_3	2,020	4.0
Th	1,735	11.5	Si_3N_4	1,900	3.1
Ti	1,680	4.4	TiO_2	1,840	4.2

Also, to provide wettability between a matrix and a fiber, a method is chosen in which an element which selectively reacts with the matrix and forms an intermediate layer is added. For example, the adhesion between the Ni matrix and Al_2O_3 fiber can be improved by adding a small quantity of Zr to the former, with the two forming ZrO_2.

Table 2. Sample Combination of Matrix and Fiber

Matrix / Fiber	Titanium	Copper	Heat-resistant alloy	Heat-resistant alloy	Superalloy
W		o	o	o	o
Mo			o		o
Ta			o		
C		o			o
B	o	o	o	o	
SiC	o				o
Al_2O_3		o	o	o	o

The difference between thermal expansion coefficients of the matrix and fiber must be taken into consideration. Since, for the movable blade of an aircraft gas turbine, heating and cooling are repeated as the turbine starts or stops operation, the matrix is separated from the fiber during this thermal cycle and deformation occurs in the fiber direction. This is the so-called ratchet deformation problem. Since it is generally almost impossible to make the thermal expansion coefficient of the matrix agree with that of the fiber, one conceivable measure is to suppress thermal ratchet deformation by raising the yield strength of the matrix. Also, a method involving the formation of an intermediate layer is being studied to ease the difference between the thermal expansion of the matrix and that of the fiber.

Since, of the various fibers, the W fiber has a particularly high melting point and is superior in creep characteristics, it is attracting attention as a reinforcement material for composite materials based on the heat-resistant alloys of Ni, Co, and Fe radicals. The W fiber is usually manufactured by cold drawing processing and has a fine processed fiber structure. As the degree of processing becomes larger, i.e., as the fiber becomes thinner, the strength increases, and a fiber of about 0.1 mmϕ is often used for a composite material due to ease of handling. The pure W alloy (undoped material) causes its crystal grains to become coarse because of secondary crystallization at comparatively low temperatures (1,200~1,400°C). To prevent this, a W fiber has been developed that raises the secondary recrystallization temperature to about 1,800°C for doped materials which have a small quantity of Al, K, and Si added, or for a solid solution containing 1 percent of ThO_2 or a small quantity (5 percent or less) of Re, and which is aimed at stabilizing processed fibers and enhancing strength.

Since the W fiber is of large specific gravity, composite reinforcement is also being studied with metal fibers of Mo, which is of comparatively low specific gravity, or ceramic fibers of C, SiC, B and Al_2O_3. Since the load stress that acts upon the movable blade of the gas turbine of an aircraft is mainly tensile stress in the direction of the blade length, because of the

centrifugal force during rotation, the strength anisotropy of a fiber-reinforced composite material can be used effectively. In other words, as shown in Figure 1, fibers can be unidirectionally laid out along the longitudinal direction of the blade, and the kind and content of fibers can be changed locally, depending on the structurally necessary strength values. Since the life of the movable blade is determined by the creep rupture life at the critical zone of the middle part, this part can be made to contain many particularly high-strength W fibers, while the blade root part, which thickens, can be made to contain a smaller number of fibers. A method for reducing the blade weight can be derived by using ceramic fibers of low specific gravity for the tip of the movable blade.

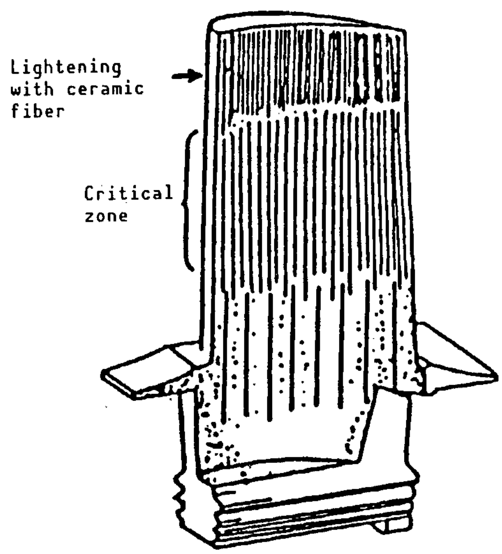

Figure 1. Example of Turbine Movable Blade Reinforced With Fibers

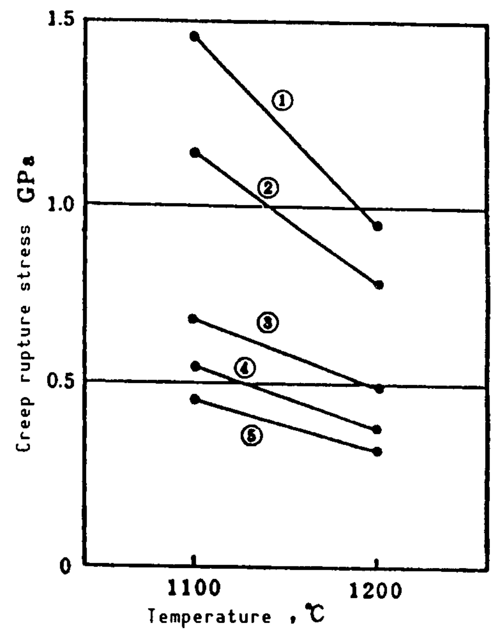

Figure 2. 100-Hour Creep Rupture Strength of Various W Fibers

Performance

Figure 2 shows the 100-hour creep rupture strength of various W-based fibers, which are expected to be used as reinforcement materials for the movable blade of the turbine, at 1,100°C and 1,200°C. Compared with the pure W fiber, W-based fibers which are solid solution-containing metals and ceramics with high melting points, are found to be superior in high-temperature creep performance.

Application Fields

Aeronautics and space, automobiles, electric machines and communications devices, and sporting goods.

Notes

None.

Others

Technical development will be promoted in the future, and individual countries are expected to apply for patents related to these materials.

Purifying, Evaluating Semiconductor for Use in Optoelectronics

43067215e Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 24-26

[Article by Electrotechnical Laboratory]

[Text] **Summary**

A high-quality material manufacturing technique not previously required has come to be demanded so that in highly functionalized and integrated semiconductor devices, not only the optical properties such as the refractive index and band gap, but also electrical properties such as the number of carriers and mobility, can simultaneously satisfy target specifications. Therefore, a manufacturing technique which introduces only minor impurities and few lattice defects and exhibits very high controllability of the crystal composition and structure is demanded. With the superhigh-speed quantum optical device which employs the quantum effect of the electrons enclosed in a super minute space, the influence of impurities and lattice defects is especially large and the above-mentioned demand is stronger. Also, in order to evaluate the composition, structure, impurities, and lattice defects of these device materials, the development of a measuring means offering high spatial resolution is indispensable.

In this research, superhigh purity GaAs, a representative semiconductor material, was manufactured by the molecular beam epitaxial (MBE) method which allows epitaxial films of the highest purity and low defect density to be manufactured, and standard samples for epitaxial film evaluation were fabricated by introducing mass-separated electrically and optically active impurities, in the form of ions, into the GaAs during crystal growth. Then, a basic data base related to impurities and lattice defects was built by performing various optical measurements.

Detailed Contents

1. Manufacturing Superhigh Purity GaAs Used Molecular Beam Epitaxial (MBE) Method

A huge quantity of information on impurities and defects of GaAs has been accumulated so far, and the data base comprised of this information has been

thought to be almost complete. However, if a GaAs epitaxial film is manufactured by the MBE method, many luminous lines that have not yet been observed (CDIBE lines) can be seen in its low-temperature photoluminescence spectrum (PL), as shown in Figure 1(c), even in undoped cases (remaining impurity density: $10^{15}cm^{-3}$ or less).

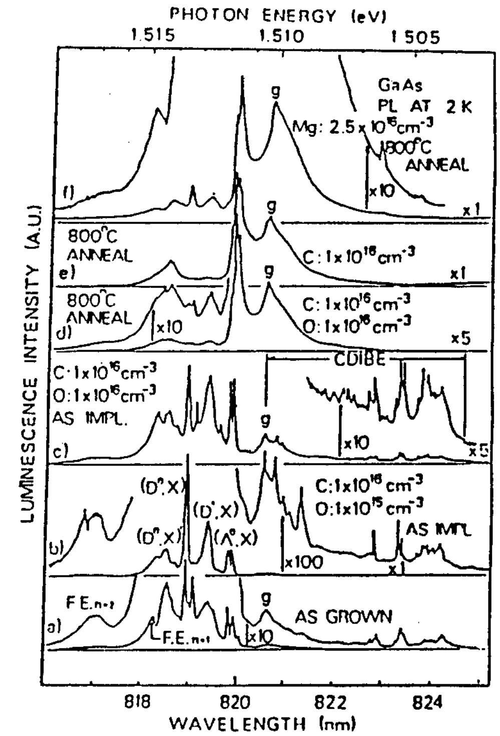

Figure 1. Photoluminescence Spectrum at 2K
(a) Case of very high purity
(b, c) PL spectrum when C ions and O ions are implanted simultaneously
(d) PL spectrum when (c) was annealed at 800°C
(e) PL spectrum when only C ions were implanted

To clarify this phenomenon, the authors recently developed an in-MBE-growth ion beam introduction device which has good mass monochromaticity, and allows diversified impurities to be introduced and various lattice defects, and evaluated samples electrically (hole effect) and by PL measurement. As a result, separate (Figure 1(e) and (f)) and simultaneous (Figure 1(b),(c) and (d)) ion implantation of C^+ and O^+ showed that the CDIBE line is composed of a pair, the C of which behaves as an acceptor atom, with O acting at an interlattice position ($C_{As}+O_{int}$) among the COs which comprise the main remaining molecules in the MBE crystal growth device. In this way, the remaining impurity gas in the MBE crystal growth device was found, for the first time, to combine with various point defects to form strong luminous centers, and the CDIBE line was determined to be usable for qualitative and quantitative judgments on remaining impurities and lattice defects. Furthermore, it was found that this CDIBE line was greatly influenced by the three growth conditions, i.e., the growth substrate temperature, crystal substrate

direction, and As4/Ga ratio, and the optimum crystal growth conditions were expected to be a 450°C or lower substrate temperature, (1,0,0) crystal direction, and As/Ga ratio near 2, at least form the standpoints of remaining superlow-density impurities and defect density.

2. Impurity Introduction Into GaAs by Using Mass-Separated Ion Beam

The authors systematically performed mainly acceptor impurity introduction experiments during or after GaAs crystal growth in a very extensive density range of from 10^{15}~$10^{22}cm^{-3}$ by the above-mentioned in-MBE-growth ion beam introduction device (Figure 2) or other methods, and observed many very strong luminous lines which had never been observed before.

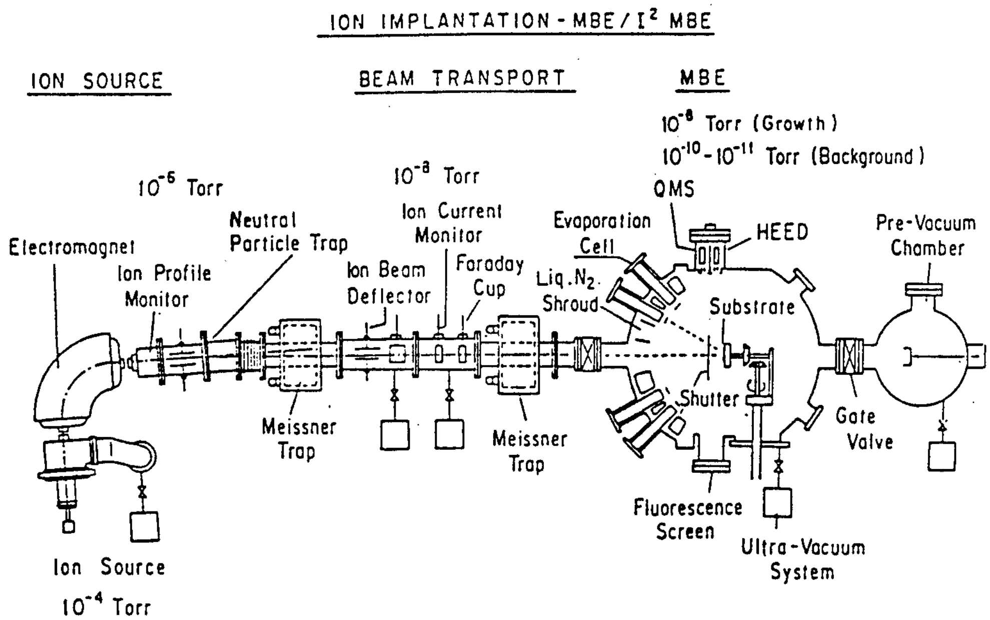

Figure 2. Conceptual Diagram of the Device During MBE Growth Ion Beam Introduction

The left half is the ion implantation device capable of 1 keV to 30 keV ion beam irradiation. The right half is the molecular beam epitaxial device. In order to match the vacuums of both devices, differential vacuum exhaust is conducted by an ion beam transport system.

Figure 3 shows examples of Mg atoms introduced by various methods. Of these luminous lines, [g-g] and [g-g]α shift to the low and high energy sides as the acceptor temperature rises. This characteristic was observed for the first time with the sample manufactured by this technique and was found to be usable for the nonelectric but optical high-space-resolution quantitative determination of acceptor density. Furthermore, in practical use, these strong luminous lines were shown to be usable for raising the brightness of semiconductor materials.[2] Simultaneous impurity ion introduction experiments performed on

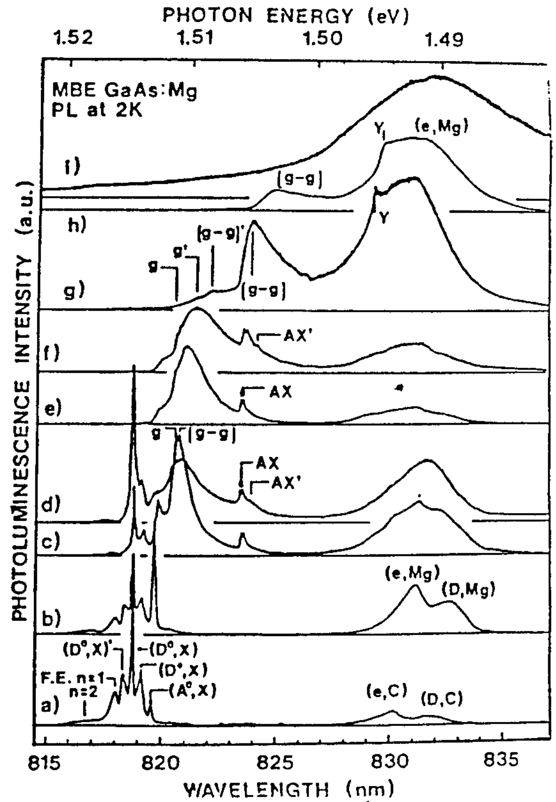

Figure 3. PL Spectrum of Mg Doped GaAs
(a) and (b) Undoped cases

donors and acceptors also showed that these many acceptor-related luminous lines (especially [g-g] disappear very easily when a very small quantity, e.g., approximately one-thirtieth of the acceptor density, of the donor (existence of optical compensation effect) exists. This was found to be the main reason for [g-g] not being observed in a sample manufactured by the conventional technique.[3] According to a detailed theoretical study, [g-g] was found to be a pair of excited acceptors, and was proved to represent an entirely new energy level in a semiconductor.[4] When multiple impurities and lattice defects exist in a semiconductor in this way, the PL spectrum differs from the simple superposition of the PL spectra caused by each impurity or lattice defect, and it has been experimentally and theoretically made clear for the first time that some luminous lines are observable only when two or more impurities and lattice defects exist simultaneously while, conversely, others disappear if two or more impurities and lattice defects exist simultaneously.[5] Therefore, in order to improve the quality of the materials for optoelectronics and build highly reliable data bases, it has been found necessary to perform experiments involving the introduction of impurities of very high mass monochromicity and lattice defects in the state where the remaining impurities and lattice defects are very few by using the in-MBE-growth ion beam introduction device, and to perform detailed optical evaluation using the PL method.

As mentioned above, it has been made clear that the previously published luminescence-related information on semiconductor materials needs to be reviewed in its entirety.

3. Building PL Data Bases for Semiconductor Materials

In order to conduct a physical evaluation of the superminute region exhibiting the quantum effect, it is indispensable that various optical evaluation methods using laser beams be developed which are capable of using superminute probe lights (observation light source for probing). The PL method, which requires no sample surface processing, is an ideal technique in this sense, but has been insufficient for quantitative discussions.

In this research, standard samples were carefully prepared by introducing diversified impurities, containing extremely small quantities of other impurities, into the sample space which is limited to an extensive density region, by using techniques 1 and 2 above. The authors also prepared samples into which accurate density-controlled multiple impurity atoms had been introduced, built a PL spectrum data base for the various impurities and lattice defects using these samples, and showed that the identification and quantification of kinds of impurities and lattice defects are possible from such information as the presence or absence of various luminous lines and phonon side band zones, the presence or absence of the above-mentioned generation and disappearance, the energy positions of luminous lines, energy half-value widths, and the relative strength of industrial luminous lines.

Performance

This in-MBE-growth ion beam introduction device is a semiconductor manufacturing device with superior controllability of composition and structure, and is capable of introducing impurities of very good mass monochromaticity in a wide density range of from 10^{15}~$10^{20}cm^{-3}$. Furthermore, this sample evaluation method can identify and quantify the impurities of various acceptors and donors which determine the capability of a device at high precision (10 percent) in the above-mentioned range.

Application

1. Evaluation of quantum device by PL method

Since this sample evaluation method is capable of high space resolution identification and quantification of the impurities of acceptors and donors which determine the physical properties of various semiconductors with an electrodeless noncontact and nondestructive technique, it will become an indispensable technique, especially for the physical evaluation of physical properties of the various quantum structures which use a superlimit minute space region.

2. Building optical data base for III-V genus compound semiconductor.

3. Manufacturing very bright light emitting element, high-speed optical switch, and modulation element

Since luminous lines such as [g-g] and [g-g]α emit light even at room temperature, this phenomenon makes it possible to manufacture a room-temperature-use very bright light emitting diode and laser diode. These luminous lines greatly change the luminescence strength and wavelength with a very weak external field (electric and magnetic fields and pressure), and their use makes it possible to manufacture a high-performance high-speed optical switch or modulation element.

Notes on Utilizing This Technique

The second-generation in-MBE-growth ion beam introduction device is currently in the design and manufacturing stages, and the use of this device will permit experiments to be conducted on the introduction of more reliable impurities and lattice defects. Also, although the conventional optical knowledge related to impurities and defects in GaAs should be entirely restudied, regarding the case of deep impurities, as well as the case in which these two impurities exist compositively, further large-scale systematic research is required.

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High-Performance Magnetooptical Material Techniques

43067215f Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 100-101

[Article by Electrotechnical Laboratory]

[Text] **Summary**

Because the functions, density and reliability of processing, storing and transmitting information have been sharply improved, the importance of optical techniques is increasing. Magnetooptical materials are strongly demanded as optical recording media which are rewritable and allow the precise and detailed control of light waves by employing the nonreciprocity and polarization characteristics unique to the magnetooptical effect. $Y_3Fe_5O_{12}$ (YIG), which has a garnet-type crystal structure, satisfies the material requirements and is a representative conventional material exhibiting superior performance in the near infrared light band. If Y is replaced by Bi, the Faraday rotation angle increases in proportion to the quantity of Bi. Therefore, high-density substitution was attempted, but $YBi_2Fe_5O_{12}$ represented the upper limit. $Bi_3Fe_5O_{12}$ (BIG), which is a garnet-type structure completely replaced by Bi in an unequilibrated phase, does not exist in nature and has never been synthesized. BIG was materialized for the first time by the epitaxial growth from the gaseous phase with the reactive ion beam sputtering (RIBS) method. BIG was found to be a superhigh-performance material exhibiting a magnetooptical performance index (F: Faraday rotation angle per unit light absorption) of as high as 55 deg/dB at a wavelength of 800 nm.

Detailed Contents

1. Magnetooptical Element and Material

In an optical circuit, the reflection on an end surface of an optical part or at a curved part of an optical path causes return light. The return light makes the departure of certain laser beams unstable at the light source. The light isolator, using the magnetooptical effect, is an important optical circuit part which cuts the return light. In addition, the circulator, which separates the input and output, the optical switch, which changes over an optical circuit, and the optical deflector, which sweeps light within a fixed angular range, can be materialized by using the magnetooptical effect.

The magnetooptical material must satisfy such conditions as 1) when the magnetooptical effect (Faraday effect) is large; 2) large absorption is weak (for optical control) or strong (for optical recording); 3) optically isotropic; 4) ferromagnetic at room temperature; and 5) chemically stable. Of the substances currently known, only the iron garnet, represented by YIG, is provided with these material conditions. For YIG, the Faraday rotation ability θ_F is +835 deg/cm and light absorption coefficient α is 700 cm^{-1} at wavelength 633 nm. To use YIG as an isolator, the deflection surface must rotate 45 deg. Therefore, the necessary light path is about 500 μm. If YIG is to be handled as an optically integrated circuit element, a further reduction by two positions becomes the target. To attain this target, the development of a material whose θ_F is larger by about two positions is desired.

2. Bi Substitution Effect in YIG

If Bi is substituted for Y in YIG, θ_F increases in proportion to the Bi substitution quantity, as shown in Figure 1. For BIG, in which Bi completely replaces the Y in YIG, a value as large as -6.2×10^4 deg/cm, about 80 times the value of YIG, is expected at a wavelength of 633 nm. Figure 2 shows that the lattice constant also increases sharply in proportion to the substituted quantity, conforming to the results of the experiment, i.e., BIG represents an unequilibrated phase and has never been either discovered in nature or synthesized artificially.

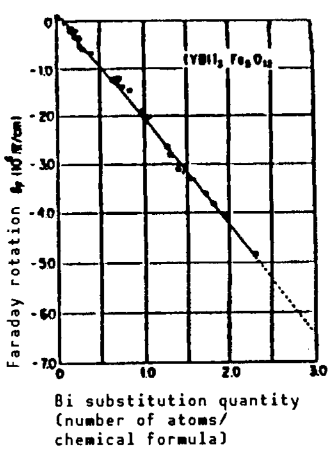

Figure 1. Dependence of Faraday Rotation Ability on Bi Substitution Quantity in YIG (Wavelength: 633 nm)

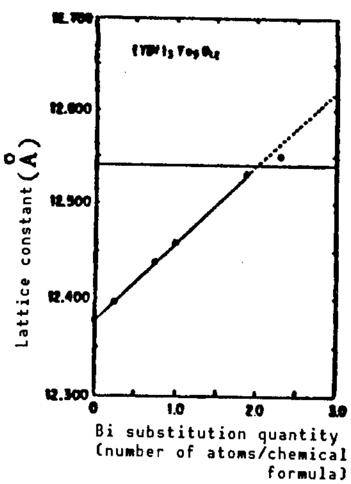

Figure 2. Dependence of Lattice Constant on Bi Substitution Quantity in YIG

3. Guideline for BIG Synthesis

The Bi-substituted YIG is usually synthesized by the liquid phase epitaxy (LPE) method. The Bi-substitution quantity increases along with increase in the density of Bi in the solution $[Bi]_M$, the increase in the growth temperature T_G, and the I in the degree of supercooling ΔTs. The increase in [Bi] and drop

in T_G increase viscosity remarkably and bring about a sharp drop in the diffusion speed. If ΔTs is made larger, the supercooled state of the solution suddenly becomes unstable, and the fine crystals in the orthoferrite phase separate. The solid solution limit composition is obtained when TG=690°C and ΔTs=150°C. These conditions can be termed the substantial limit for crystal growth being possible in the solution. As guidelines for the materialization of the BIG phase, the following can be considered: 1) separation of the reaction field and substance supply source; 2) assistance in and stabilization of garnet phase nucleus formation; and 3) epitaxial growth during accumulation. Concretely, 1) performing substance transportation in the gaseous phase rather than in the liquid phase, 2) using a single-crystal garnet substrate which permits lattice matching, and 3) keeping the substrate at an appropriate temperature and partial pressure of oxygen, are kept in mind.

4. Reactive Ion Beam Sputtering Method

The ion beam sputtering accumulation method irradiates ion beams of inactive gases, such as Ar and N_2, to the target containing component elements of the substance which is the target for accumulation, and transports the substance sputtered from the target to the substrate in the gaseous phase. When an oxide film is to be formed, gaseous or ionized oxygen is supplied and made to react during accumulation. This is called the reactive ion beam sputtering (RIBS) method. Figure 3 shows the concept. In the RIBS method, the substance supply source (target), transportation means (ion source), and accumulation, reaction and synthesis portions (substrate) are independent of one another.

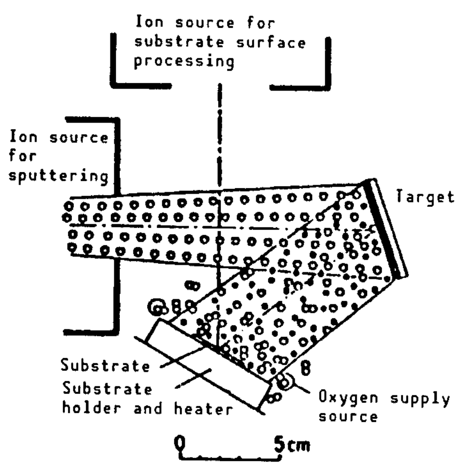

Figure 3. "Concept of Reactive Ion Beam Sputtering Accumulation Method"
o Ar ion; • Sputtering particle; oo Oxygen gas

The energy of the spattered particles is distributed, peaking near several eV. This energy is suitable for accumulation and has no etching effect. However, it corresponds to several ten thousand degrees if converted into heat energy and has the effect of promoting the surface diffusion of accumulated particles and a crystallization reaction. Compared with the high-frequency sputtering method, the RIBS method is higher by two positions or more in degree of vacuum

and does not expose the substrate to plasma. If these characteristics are taken into consideration, the RIBS method can be said to be a promising measure for realizing the above-mentioned BIG synthesis guideline.

5. BIG Synthesis

The BIG fabrication procedure is as follows: 1) Sputtered particles are accumulated on a single crystal garnet substrate with a high lattice constant by irradiating Ar ions onto a mixed ceramic target containing Bi_2O_3 and Fe_2O_3. 2) During the accumulation, the substrate is kept within an appropriate temperature range, and the vicinity of the substrate is kept under an appropriate oxygen particle pressure. 3) The degree of vacuum is retained at the 10^{-5} Torr level.

Performance

BIG is obtained as an epitaxially grown single crystal thin film according to the direction of the substrate used. The crystal structure is the direction of the substrate used. The crystal structure is the optically isotropic garnet type. The magnetic transition temperature is 500 K or higher. It is ferromagnetic at room temperature. The measured value of the lattice constant agrees with the calculated value of the lattice constant agrees with the calculated value of 12.624 A. At a wavelength of 633 nm, θ_F is -7.2×10^4 deg/cm and α is 1.6×10^3 cm^{-1}. The K_{err} effect, the magnetooptical effect for reflected light, shows a value of as high as −1.1 deg at a wavelength of 465 nm.

Application Fields

(1) Optical isolator: Indispensable for the departing wavelength stabilization of the 800 nm-band wavelength short-wavelength DFB semiconductor laser. The large demand for the short-wavelength semiconductor laser is expected for optical disks and short-distance optical networks. An appropriate isolator is not yet available for the 800 nm band.

(2) Optically integrated circuit: Waveguide-type magnetooptical elements of the 10 μm order size can be formed for the 800 nm band. Integration with semiconductor elements materializes optical electronic integrated circuit [OEIC], and high performance and high reliability are attained in communications and information processing.

(3) Optical recording media: Rewritable optical recording is the main current in large-capacity recording. Since the current noncrystalline metal media have small magnetooptical effect and pose a problem in chemical stability, oxide films are being thought of as promising next-generation media.

Related Patents

Patent applied for 1987-247452 "Ferromagnetic compounds and their thin film manufacturing method"

Patent applied for 1987-253063 "Method for target manufacturing"

Research on Light-Reactive Materials Using Molecular Layout Field

43067215g Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 102-103

[Article by Chemical Engineering Laboratory, Tsukuba]

[Text] **Summary**

Langmuir-Blodgett film (LB film), which is a superthin organic film, can arbitrarily accumulate monomolecular films on a water surface. Since it permits a laminated structure design of organic molecules at the molecular level, it is attracting attention in extensive areas, from basic research to application areas. The optochemical reaction in LB film is of particular interest from the viewpoint of various optoelectronics-related application techniques, such as photoresists, optical memory, and optical modulation elements.

In this research, the authors fabricated an LB film which includes coloring matters (azobenzenes) in the empty holes of a bilyophilic long-chain alkyl β-cyclodextrine derivative (CD) and examined its optical reactivity. This was the first example of applying a host-guest compound to the LB film. In this LB film, coloring matters are included in the CD holes and the optical isomerizing reaction advances reversibly. Therefore, this host-guest LB film is significant in that it is fabricated without chemically modifying the long-chain alkyl radical to the coloring matter, and that it can be developed as a superthin film having the free volume which allows reversible reaction isomerization.

Detailed Contents

1. Fabrication of Host-Guest LB Film

The LB film is a laminated multilayer film obtained by compressing the bilyophilic compound developed on a clean water surface as a monomolecular film to form a condensed film and moving it onto a solid substrate. Several research efforts have been performed so far on LB film containing chromophoric groups having photochromic functions. In order to fabricate a reactive LB film, however, the bilyophilic property must be given to the film-forming molecules. Therefore, the chromophoric groups must be chemically modified

such as through long-chain alkyl and polarity radicals. Also, since the optical isomerization does not advance toward increasing the molecule-occupied area in an LB film in which the coloring matters are densely filled, the development of an LB film which materializes an optical isomerization reaction by which the molecule-occupied area reverses represents an important subject for examination.

The authors examined the fabrication of an LB film in which azobenzenes were included in the empty holes of the long-chain β-cyclodextrine derivative, and confirmed that the host-guest film had been fabricated, with the polarized light UV spectrum of the LB film and its measurement included in the chloroform solution. That is, this LB film is the first example of a host-guest compound application, and cyclodextrine is uniformly oriented in the film. Therefore, it is a superthin film in which chromophoric groups without alkyl chains have been included n the empty holes which are regularly arranged and have the same volume as in an LB film. To fabricate this LB film, the chloroform solution containing the CD including coloring matters was dropped on a twice-distilled water surface and accumulated on a quartz substrate which had been subjected to hydrophobic processing after compression at a surface pressure of 30 mN/m. Also, the host-guest LB film was obtained by dissolving coloring matters in water in advance and making the solution form include complex radicals on the air-water boundary surface.

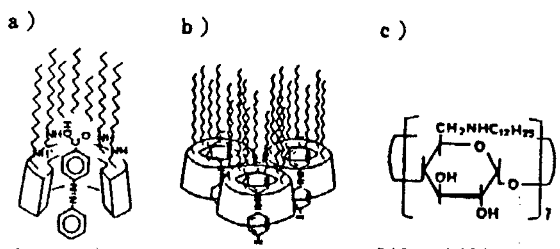

Figure 1. Host-Guest LB Film

2. Optical Isomerization Reaction of Host-Guest LB Film

The authors measured the absorption spectrum change of intentionizing (sic) reaction in the LB film using an ultraviolet-visible self-recording spectrophotometer. The cross illumination method was used and the quartz substrate was set in the sample room so that the exciting light and monitor light were mutually incident at 45°.

Figure 2 shows the absorption spectrum change for each exciting light irradiation when phenyl azobenzoic acid (PAB) was used as the coloring matter. The absorption spectrum before irradiation agrees with that of the transbody PAB in chloroform solution, except for the absorption increase by CD on the short wavelength side. If light of 36 nm is irradiated, absorption at the 228 nm and 327 nm absorption bands decreased, while that at 255 nm and 445 nm increased. This absorption spectrum change closely resembles the optical isomerization reaction from the transbody to the cisbody in the chloroform

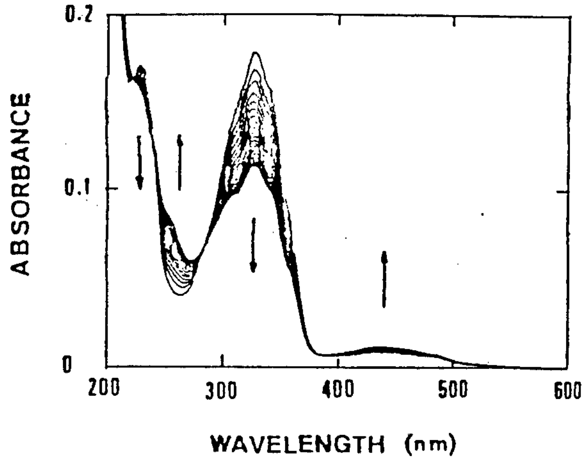

Figure 2. Trans–cis Optical Isomerization Reaction of LB Film

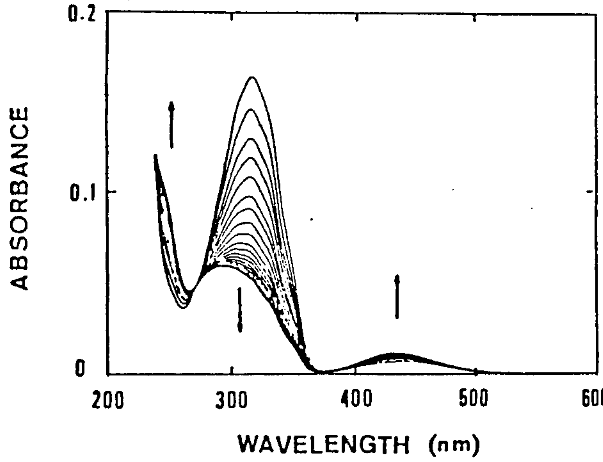

Figure 3. Trans–cis Optical Isomerization Reaction in Chloroform Solution

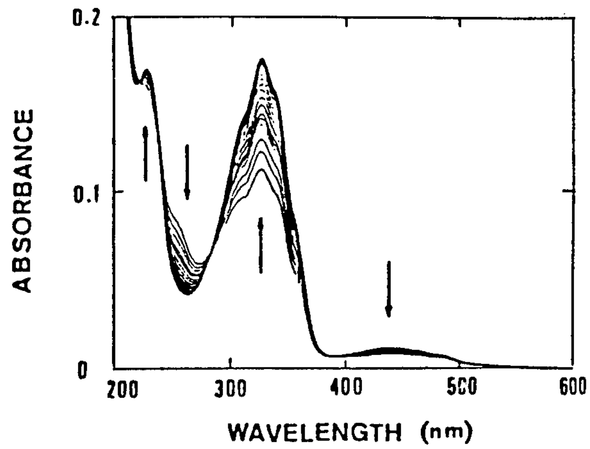

Figure 3. Trans–cis Optical Isomerization Reaction of LB Film

solution shown in Figure 3. Therefore, authors thought that similar optical isomerization occurred in the LB film. Also, when light of 254 nm, corresponding to the absorption body of the cisbody, was irradiated to a CD-PAB LB film after an optical stationary state had been reached by irradiating light of 360 nm, the reverse spectrum change to that of Figure 2 occurred, as shown in Figure 4. This shows the optical isomerization reaction from the cisbody to the transbody. This isomerization occurred in the same way when light of 400 nm, which is the n–π^* absorption band of the cisbody, was excited. Therefore, selective irradiation of lights of 360 nm and 254 nm (or of 400 nm or a longer wavelength) could reversibly cause a trans–cis isomerization reaction in the LB film. In addition, the repeatability of the isomerization reaction was also good.

It was found that with other azo coloring matters (p–methylred, methylorange), the host–guest LB film could be fabricated in the same way as with PAB, and optical isomerization was reversible.

Performance

Since molecules are densely filled in the LB film fields in the research which has been conducted so far on the optically reactive LB film using bilyophilic compounds, the optical reaction has been said not to advance in the direction of the molecule-occupied area. In causing reversible operation isomerization, therefore, the molecular design of the optical reaction in the LB film did not advance smoothly because the method of mixing soft molecules, such as tripalmitin, in the LB film was used. However, the problem has been greatly improved by the results obtained by the authors.

A superior performance of the new optically reactive LB film which uses the cyclodextrine derivative and has been made to contain azobenzene by applying this host-guest mutual reaction involves the isomerization reaction occurring reversibly at a high conversion rate and with good repeatability. Such high-performance optical isomerization behavior is thought to occur because the empty holes of the cyclodextrine derivative provide sufficient free volume for the isomerization of azobenzenes. The LB film has come to be made from many other photochromic molecules by including them in cyclodextrine, using the host-guest mutual reaction without using shared combinations.

Application Field

Since one coloring matter corresponds to one bit of memory, and various coloring matters can be accumulated multistepwise in the LB film to form a superthin film, the LB film is expected to be applicable to high-density multiplex optical memory, optical modulation elements, and high-resolution photoresists.

Related Patents

Patent applied for: "Cyclodextrine derivative, organic superthin film using it, and its manufacturing method"

Patent applied for: "Organic superthin film of a material including coloring matter and cyclodextrine derivative and its manufacturing method"

References

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2. Yabe, A., et al., Ibid., 1988, pp 1-4.

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Expert System for Patent Act

43067215h Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 165-166

[Article by Electrotechnical Laboratory]

[Text] **Summary**

As a computer application, systems (expert systems) which are provided with experts' knowledge and perform diagnostics, forecasting, and action plans are being actively developed. Practical expert systems have been developed for medical diagnostics, machine troubleshooting, and design support, but few are used in the legal field due to the difficulties encountered in legal interpretation and representation and the use of legal knowledge. This research is mainly aimed at the representation and use of knowledge and the development of new techniques, the development of the knowledge representation language KRIP/L, and the creation, on a trial basis, of the patent law expert system KRIP as an application of the language. Legal knowledge has been classified into several categories, and appropriate representation and usage methods have been provided for each category. If KRIP/L is used, legal knowledge which tended to be complex with the conventional technique is represented in a form near to intuition, and further, experts' strategic knowledge and changes in situations can be represented in a natural form. Also, the patent act expert system KRIP has a function by which, if a simple case regarding patent application is given, it describes the related matters that have occurred and advises the next procedure that should be followed.

Detailed Contents

1. Knowledge Representation Language KRIP/L

As methods of representing knowledge using a computer, frames, logical expressions, production rules, and networks are available. Since legal provisions are written logically, comparatively many systems have been written with logical expressions, but the representation tends to become complex if additional knowledge that does not appear in the provisions is also described. Therefore, the authors have developed the knowledge representation language KRIP/L which merges objects and extended Prolog (logical expression).

The object is a calculation unit consisting of a group of data and a group of procedures related to the data (called the method). In conventional programs, the program and data are independent of each other, and calculation is executed if data is provided to the program. A program based on the object consists of several objects and calculation is performed by sending instructions (messages) to an object. The object to which the message is sent activates a method based on the message contents and manipulates its own data.

Extended Prolog has been obtained by extending the logical language Prolog so as to be able to represent the status changes and aspects (can do, must do, and must not do) that accompany the passage of time. For example,

[A:B] = > P:-Q.
[C:D] → may R: -S. means "if Q materializes while A and B occur, P materializes," and "if S materializes while C and D occur, action R is legal," respectively.

KRIP/L can summon extended Prolog from an object or read the object contents from extended Prolog. The knowledge representation by KRIP/L represents individual concepts with objects and makes natural representation possible by representing general rules with extended Prolog.

2. Patent Act Expert System KRIP

The patent act is a law that is related to the procedures for obtaining a patent and the accompanying rights. For individual procedures, the conditions for performing them and their effects are regulated.

As shown in Figure 1, patent act knowledge is described by KRIP/L. The concepts that appear in the law (person, application procedure, right, and action) are represented as individual objects, and when provisions related to the concepts exist, the instruction "Examine the provision" is described in an object as a method. Provisions are described by using extended Prolog. Relationships between concepts, priority relationships between provisions, and strategic knowledge of provision applications, are described in an object. Experts' knowledge, examination standards, and interpretation changes based on precedents are also described in an object.

The main functions of KRIP are diagnostics, forecasting, and advice. If there is a case to consult with, it is first represented in a group of objects and is stored in the fact data base. For example, if the case:

1 June 1984	Mr Yamada invented superconductive substance X and its manufacturing method Y.
2 July 1984	Mr Yamada completed patent application, with X written in the "detailed explanation" and Y written in the "request range."
8 August 1984	Mr Yamada requested application examination.
7 January 1986	The application was made public.
1 September 1987	Mr Yamada received a refusal reason notification based on Act Provision 36 Clause 4

is input, five objects are written into the fact data base.

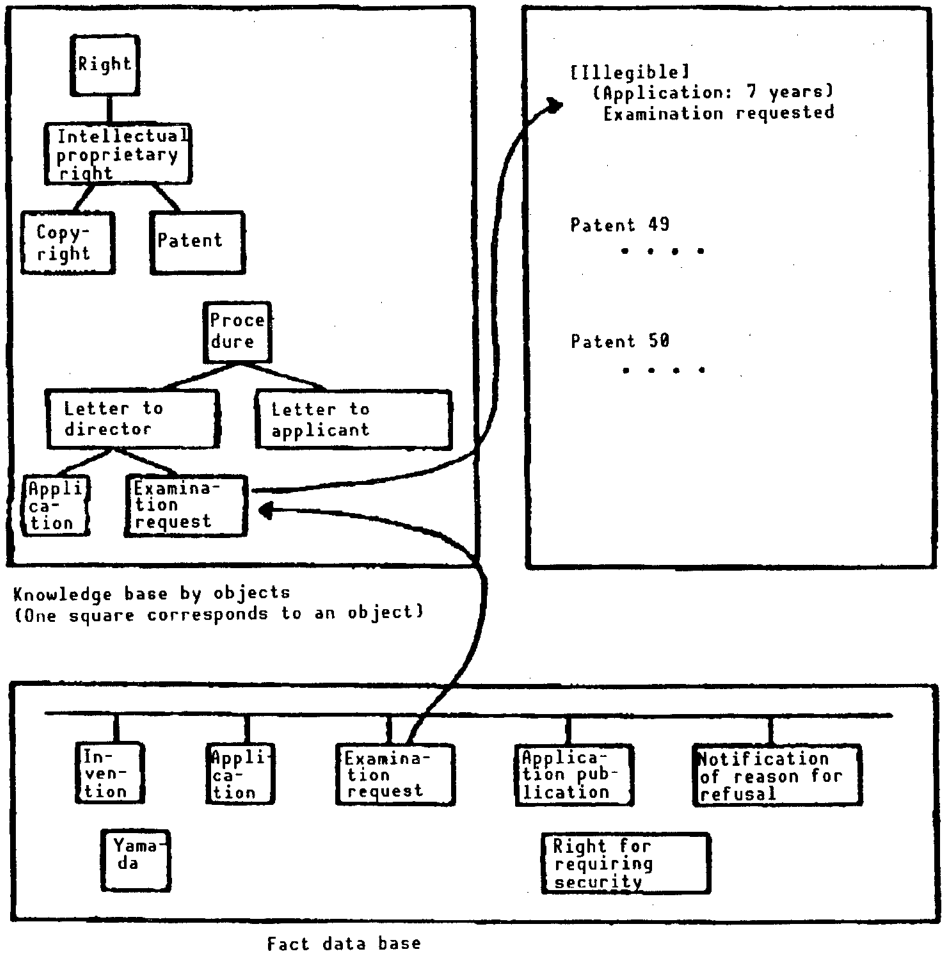

Figure 1. Representation of Patent Act by KRIP/L

If the system is activated, it sends messages to the objects in the fact data base, beginning with the oldest one. The object receiving the message retrieves the related provision using the method written in the corresponding object in the knowledge base, verifies the legality, and simulates the effects.

The object explains what right has occurred, discovers illegal procedures, and advises the procedure that Mr Yamada should follow. Also, the procedure format and charge are displayed.

Performance

1. Knowledge Representation Language KRIP/L

KRIP/L is suitable for describing the status changes that accompany the passage of time, and is usable for the development not only of expert systems for law, but also those in other fields. This system consists of four support modules for information display during knowledge input and execution, and is usable as a so-called general purpose AI tool.

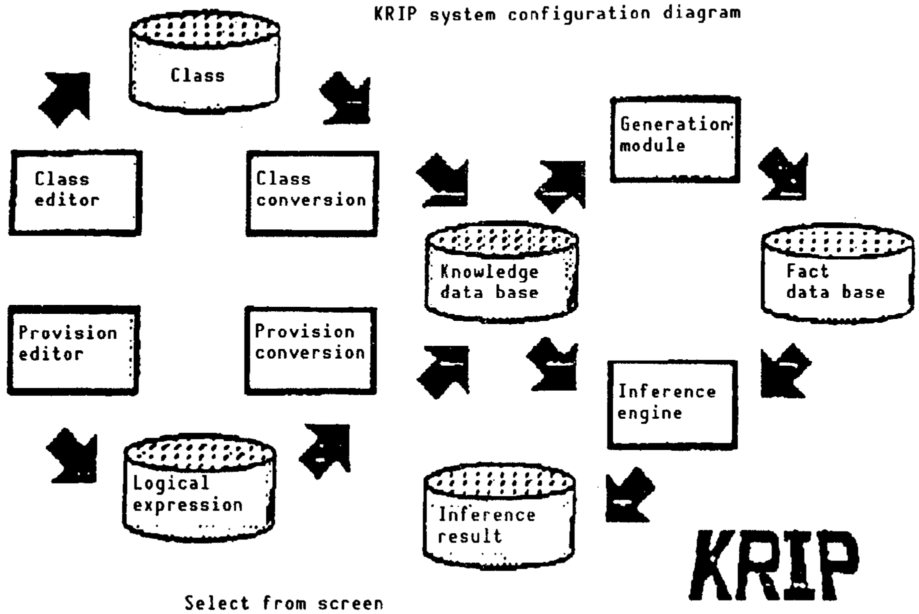

Figure 2. KRIP/L System Configuration

2. Patent Law Expert System KRIP

The personal computer version and Prolog machine (PSI) version are available. The former is generated by KRIP/L, but the latter is generated by ESP, a similar language. No difference exists between the functions of these two tools, but the scale of the former has been reduced because of insufficient memory.

The main functions of this system are diagnostics and providing advice, but the system can compare two procedures or rights (common and different points) and retrieve a provision from the keyword.

Since this system analyzes provisions in detail and its technical problems have been fairly well resolved, as compared with the conventional method, it has the possibility of being put to practical use. However, since the main purpose of this research is not the development of expert systems themselves but the development of knowledge representation and inference techniques instead, the expert systems already materialized are limited to the main provisions of the Patent Act.

Application Fields

The KRIP/L system can also be used as a general-purpose AI tool, and is especially suitable for fields in which situations change with the passage of time.

The law representation method that was used in KRIP system development can also be applied to the development of expert systems for other laws.

Simple Robot Driving Techniques Using Visual Recognition Processing

43067215i Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 169-170

[Article by Government Industrial Research Institute, Shikoku]

[Text] **Summary**

Visual recognition processing techniques are in demand in the industrial world for practical use in the rationalization and automation of various tasks. Along with enhancing the processing ability of the personal computer itself, various high-performance image processing boards have been put on the market and larger capacity RAM memories have become available. Having studied the techniques necessary for building and driving a simple robot system with a visual recognition system by combining these commercial devices, this article attaches importance to the techniques which perform visual recognition processing from the image information input from a CCD camera, identify the position of the object body from the results of the processing, and drive the robot arm.

Application Fields

Techniques for image processing, such as visual recognition processing, are indispensable for various intelligence work and are applicable to a very extensive field.

Detailed Contents

1. System Configuration

Figure 1 shows the configuration of this system. The personal computer PC9801VM (384 KB, 10 MHz) was used as the controller, while the color NTSC signal digitizing image memory board (Video 88, made by NBC) was used as the image receptor board. This board is inserted into a slot of the personal computer when used, takes in 256 x 256 pixel data from NTSC signals in a tone of 6 bits for the individual colors R, G and B (red, green and blue), and its frame memory can be freely accessed as internal memory.

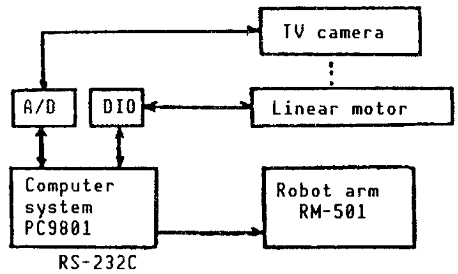

Figure 1. Hardware for Simple Robot System

For image input, a color CCD camera attached to the linear motor (Oriental brand) was used, and three-dimensional vision was obtained by driving the linear motor from the personal computer. A small multijoint robot (MOVEMASTER-11, made by Mitsubishi Electric Corp.) was used as the robot arm and driven by the personal computer via RS232.

2. Discrimination of Object Body

Some object bodies are of unique colors which are easy to discriminate from the surrounding environment and can be recognized comparatively easily in some cases if color information is used. This system adopts binary representation to extract only a specific object body using color information, reducing the time for recognition processing, and identifies the object body using the area of a region in binary representation and characteristic information, such as shapes. The object image is first classified into 8~16 clusters by the ISODATA method, which is a non-tone clustering (set classification) technique, a man then selects the work object from the classified clusters, and the deviation values of the cluster are used as the color information for binary representation. Clustering is performed on a total of 64 x 64 pixels. The work time necessary is about 1 minute for the classification up to 8 classes and about 2 minutes for the classification up to 16 classes. A pseudo color display of the classified results is also made on the personal computer's CRT.

3. Three-Dimensional Vision and Matching

In order to grasp a specific object body, accurate three-dimensional information must be calculated. Three-dimensional information can be calculated by observing the object three-dimensionally and performing the matching processing to obtain the corresponding points of the left and right images. This system mainly uses the matching method, which makes correspondences for each area, reducing the calculation time. To prevent false correspondence, corresponding points are obtained via the three visual point stereo image from the site of the object's center of gravity, while characteristic values of the shape are found by driving a camera with a linear motor and taking in the image from three different points, as shown in Figure 2. Figure 3 shows the matching sequence. Taking in the image and driving the linear motor are both performed automatically, and the processing from the first image taking to the determination of the last three-dimensional information terminates in about 4 seconds.

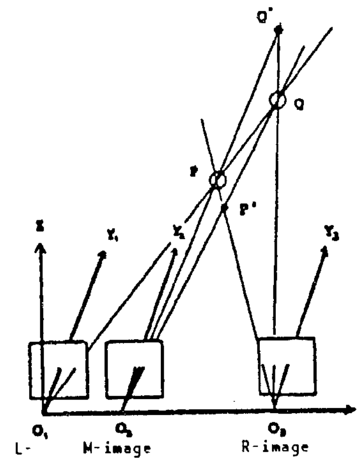

Figure 2. Principle of Three Parallax Stereo Method

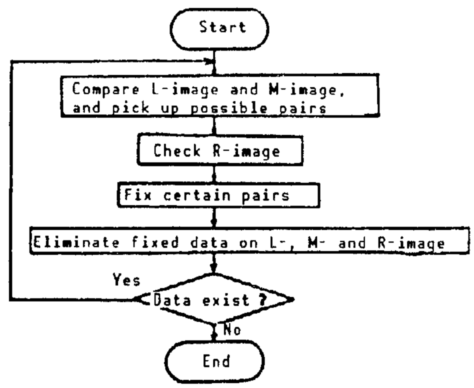

Figure 3. Sequence for Obtaining Object Correspondence for Left and Right Images

When correspondence is obtained for each area, especially, when areas are extracted by binary-represented color information, as is done in this system, the superimposition of the object body merges areas, and the characteristic values of the center of gravity and shape of the object body are recognized individually from the actual values. Since this system takes characteristics of the area shape into consideration when determining corresponding points, it determines that no corresponding points exist when occlusion (superimposition) exists. When the existence of the object body itself has been confirmed, but no corresponding points can be found, the routine (a program) for obtaining correspondence by matching each point is activated. In this routine, work is performed as a two visual point stereo operation which uses the image data obtained at both the left and right ends. Of the left and right images, the image in which the object body is completely separated is handled, the RGB brightness distribution in the area of the rectangle circumscribing the object body which has been determined to exist nearest the camera is made standard, correlation processing is performed on the image data obtained at the opposite position, and the position of the center of gravity of the corresponding area is obtained. If the correspondence relationship is obtained by correlation

processing, the three-dimensional position is determined by the principle of triangulation employing the information about the centers of gravity of the left and right end images.

4. Driving Robot Arm

The small multijoint robot used in this experiment has five degrees of freedom and its structure is shown in Figure 4. The robot arm is driven by obtaining the value actually instructed to it using calculated values from 01 to 05 and the current robot arm posture conditions.

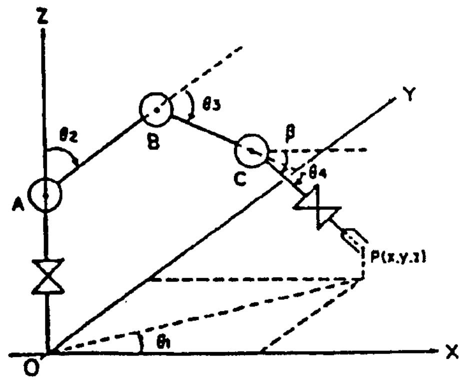

Figure 4. Outline of Simple Robot Hand MOVEMASTER

As the results of an experiment in which red, blue, yellow and white pingpong balls were laid out in space at random and only the white pingpong ball was to be selected, in about 20 seconds this system could recognize the object body and also could grasp the object body by moving the robot arm. In this operation, the recognition of the object body and the calculation of the three-dimensional information terminated in about 4 seconds, with most of the time spent in moving the robot arm.

Notes, Etc.

Since the program for the visual recognition and processing portion has been modularized, each module can be used as required.

Natural Language Syntax, Meaning Analysis Technique

43067215j Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 176-177

[Article by Electrotechnical Laboratory]

[Text] **Summary**

The language used by man is called natural language as opposed to the programming languages used by computers. The authors are conducting research aimed at building theories and systems for natural conversation between man and computer, as well as the automatic understanding and storing of textual contents. In this research, the authors developed a technique to analyze the syntax information using grammar rules and the semantic information in one sentence as basic research aimed at materializing a language function similar to that of man. For the former, the syntax analysis technique, a complete model search method, which is the fastest and can take all possibilities into consideration, was developed. For the latter, the authors developed a semantic analysis technique, i.e., an analysis method employing a concept dictionary that does not depend on a specific language either to eliminate the ambiguity occurring in syntax analysis by using the meaning of the word or to extract the semantic structure centered around the verbs in a sentence, and materialized a highly expansible semantic analysis system.

Detailed Contents

1. Syntax Analysis Technique

Since, unlike English, Japanese has no gap between words, the ambiguity that accompanies when words start has been an important subject for study. In this research, the format of the word dictionary (a dictionary which includes indexes of words and information on categories as well as on practical word usage) has been improved so that all cutout possibilities can be extracted when the syntax analysis system consults the dictionary. This technique has been named the complete model search syntax analysis method.

In the extraction of the syntax tree, which is based on grammar rules, the right branch structure has been adopted and a technique has been developed that enables the ambiguity at the syntax tree level to be sent to the semantic

analysis system without dropping information. The conventional method grasped all the modification and reception structures between two clauses as differences between syntax tree types, creating a syntax tree whose number of modifications and reception ambiguities could reach the number of items squared (Figure 1). Therefore, a slightly longer sentence often contained more than 100 ambiguities, posing a large problem. As shown in Figure 2, however, the authors' new technique has a structure which allows a noun clause (pp) and verb clause (sent) to modify any part on the right, and is arranged so as to be able to represent sufficient syntax information with one analysis tree.

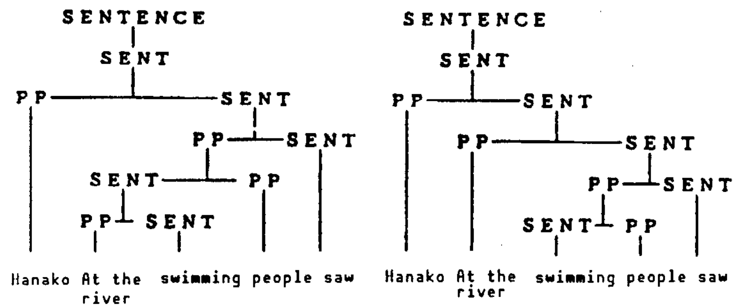

Figure 1. Two Syntax Trees Showing Ambiguity of Modification and Reception of "At the River"

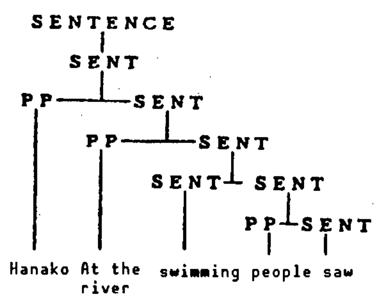

Figure 2. Right Branch Structure of Syntax Tree

Also, the algorithm for applying grammar rules is a very efficient method which merges the forecast drive type and data drive type, making real time processing possible.

2. Semantic Analysis Technique

The configuration elements comprising the semantic analysis algorithm are the semantic analysis algorithm that accompanies the grammar rules and the semantic description described in each dictionary.

The semantic analysis algorithm starts from the syntax tree sent from the syntax analysis system and puts forward a semantic algorithm, starting from the bottom right element and proceeding to the left in the right branch structure (Figure 2). In other words, this makes "who," "where," "when," "what," and "did" clear.

In the case of Japanese, the combination of a verb and a case or a prepositional particle forms the base, and correspondence between a verb and "what" or "where" is searched as semantic analysis. From the combination of "swim" and "at," for example, the possibility that the noun "river" existing before "at" corresponds to "swimming spot" is detected. Therefore, if the item "swim" in the concept dictionary is looked at, the limiting information "place where there is water" has been described as "place." Therefore, the item "river" in the concept dictionary is looked at, "place where there is water" is found in the upper concept of "river," and the limitation has come to be satisfied. In this way, "river" enters the "place" of "to swim 1," as shown in Figure 3.

To see 1		To swim 1	
Concept	To see	Concept	To swim
Actor	Hanako	Actor	Person 1
Object	Person 1	Place	River
Aspect	Completion	Aspect	Continued action

Person One	
Concept	Human

Figure 3. Meaning (Semantic) Analysis Results (Corresponds to Figure 2)

Also, since "animal" exists as a limitation of the actor of concept "to see" and "Hanako" is a lower concept of "animal," "Hanako" enters the actor of "to see 1." With a similar algorithm, the semantic analysis results shown in Figure 3 are obtained. The concept dictionary is a dictionary describing concept names, lower concepts, higher concepts, and limiting conditions, and adopts a representation method independent of individual languages such as Japanese and English. If the upper and lower conceptual relationships are used, much related information becomes possible to store and retrieve effectively. If "bird flies" is described, all the lower concepts of "bird" come to have the attribute "fly," and there is no need to write "fly" for each lower concept. The Japanese concept correspondence dictionary makes Japanese nouns and verbs correspond to concepts. As mentioned earlier, for example, in the case of the combination "Hanako" "(subject marker)" "sees," the Japanese concept correspondence dictionary provides the information that, with the verb "to see," the word immediately preceding "(subject marker)" becomes the actor of the concept "to see."

Performance

1. Syntax Analysis System

The complete horizontal search syntax analysis algorithm developed in this research is the most efficient of the existing techniques and, in addition, allows all possibilities to be comprehensively searched. Even on a workstation, the processing speed is sufficient to be practical.

This syntax analysis technique has been developed with Japanese as the nucleus, but if an English dictionary and grammar are prepared, it can easily be expanded to English.

2. Semantic Analysis System

Since semantic information is classified and described in three dictionaries, i.e., the Japanese dictionary, Japanese concept conversion dictionary, and concept dictionary, the perspective is good and the expansibility is high.

The current system has materialized basic functions, but is aimed at high-speed operation with the algorithm for retrieval of the concept dictionary and has sufficient performance for handling the expansion of Japanese vocabulary.

Application Fields

Machine translation: Directly useful, especially for the machine translation of an intermediate language system using the concept dictionary.

Context analysis: Applicable as the basic technique for context analysis in the next stage of semantic analysis.

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Synthesis of $SiC-Si_3N_4$ Composite Fine Grain Powder

43067215k Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 264-265

[Article by Government Industrial Development Laboratory, Hokkaido]

[Text] **Summary**

Ceramic superfine particles, such as those composed of SiC and Si_3N_4, are attracting attention as sintering materials. Although these superfine particles are being synthesized by a gaseous reaction, such as the high-frequency plasma method, the authors tried to synthesize SiC superfine particles using a carbonic acid gas laser as basic research for the manufacturing of $SiC-Si_3N_4$ composite fine powder. In some cases, silane (SiH_4) was used as the raw material gas for ceramic superfine particle synthesis using the carbonic acid gas laser. However, since SiH_4 is chemically unstable, difficult to handle, and expensive, the authors used dichlor silane (SiH_2Cl_2), which was easier to handle and less expensive than SiH_4.

Detailed Contents

1. Experiments

(1) Experimental device

Apollo Inc.'s tunable CO_2 laser model 150 (maximum output: 150 w) was used as the carbonic acid laser. Figure 1 shows a rough sketch of the experimental device. A mixture of dichlor silane and ethylene, the reaction gas, was made to jet from a stainless steel tube with an internal diameter of 1 mm, and a reaction was caused by irradiating a laser beam perpendicularly to the gas stream. Using a lens, the beam system converted the approximately 10-mm diameter laser beam into about 2 mmϕ. The wavelength of the laser beam used was 10.6 μm and its power density was 3.66 kw/cm^2.

(2) Experimental method

First, the inside of the reactor was made a vacuum and replaced by Ar gas. Then, the reaction gas was introduced into the reactor and reaction was caused by irradiating a laser beam. All the experiments were performed at

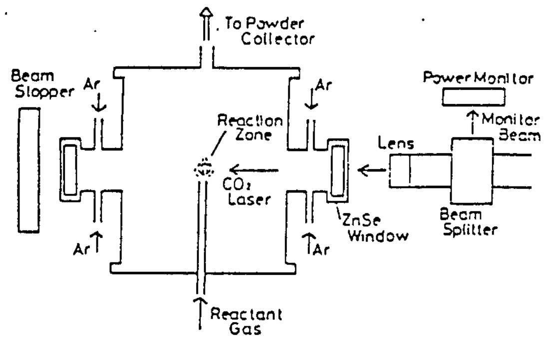

Figure 1. Experimental Device

atmospheric pressure. The generated powder was collected by precipitating it in a collection bottle. The temperature of the reaction flame was measured with a pyroscope.

For the powder, the crystal structure was observed by X-ray diffraction, the specific surface area by the BET/point method, the entire C quantity by the Coulomb measurement method, and the particle shape by a transmission electron microscope (TEM). Also, since the generated powder had a strong hydrochloric smell, it was dried in an N_2 current at about 100°C for 2 hours or more until the hydrochloric smell had diminished.

2. Results and Discussion

Table 1 shows the manufacturing conditions, reaction flame temperature, specific surface area, C-Si mol ratio, and results of X-ray analysis for the powder.

Table 1. Experimental Results

Sample No	SiH_2Cl_2 (sccm)	C_2H_4 (sccm)	Reaction zone temp.(°C)	Specific surface area (m^2/g)	C/Si molar ratio	XRD	Color
1	4	15	---	57.4	6.13	C,β-,α(VI)-SiC	black
2	10	0	<1000	14.1	----	Si	brown
3	10	3	<1000	44.9	0.55	β-,α(VI)-SiC,Si	dark brown
4	10	5	>2800	153.3	0.64	β-,α(VI)-SiC	dark grey
5	10	8	>2800	111.8	1.10	β-,α(VI)-SiC,C	"
6	10	10	>2800	78.1	1.87	C,β-,α(VI)-SiC	"
7	10	13	>2800	67.0	2.64	"	"
8	10	15	>2800	72.9	3.07	"	"
9	20	5	2550-2660	104.9	0.51	β-,α(VI)-SiC	brown
10	20	10	2300-2420	79.8	0.81	"	grey
11	20	15	>2800	88.1	1.34	β-,α(VI)-SiC,C	dark grey
12	30	15	1840-1920	86.1	0.74	β-,α(VI)-SiC	grey

These results are discussed as shown below.

(1) As a result of X-ray analysis, this powder is thought to be a mixture of β-SiC and α-SiC. Also, when the C_2H_4/SiH_2Cl_2 ratio was small, the Si diffraction line was observed, while when the ratio was large, the C diffraction line was observed.

(2) The C/Si ratio of the powder changed along with the C_2H_4/SiH_2Cl_2 ratio, resulting in the C/Si ratio reaching 1 when the C_2H_4 content exceeded that anticipated from $SiH_2Cl_2 + 1/2C_2H_4 \rightarrow SiC + 2HCl = 2H_2$.

(3) The specific surface area tended to become smaller as the C_2H_4 flow quantity decreased.

(4) According to electron microscope observation, particle shapes were generally nearly spherical. If the C/Si ratio exceeded approximately 0.6, the particle with radiating projections shown at the bottom left in Photograph 1 [not reproduced] was seen. The composition and growth mechanism of these radiating particles require further examination.

(5) The reaction flame temperature measured with a pyroscope varies with the entire reaction gas flow quantity and C_2H_4/SiH_2Cl_2 flow quantity ratio. If the entire gas flow quantity becomes larger, the flow speed increases and, since the passing time in the laser beams becomes shorter, the reaction flame temperature decreases.

Also the reaction flame temperature was 1,000°C or lower when the C_2H_4/SiH_2Cl_2 flow quantity ratio was 0.3 or less, but reached 1,800°C or higher when the C_2H_4 flow quantity increased. This is because $SiH_2Cl_2 \rightarrow Si + 2HCl$ (ΔH = 31 kcal/mol, 1,000°C) is an endothermic reaction while the reaction $1/2C_2H_2 \rightarrow C \div H_2$ (ΔH = −4.6 kcal/mol, 1,000°C) is an exothermic reaction, and if the C_2H_4 flow quantity increases, the reaction flame temperature is raised by the contribution of the exothermic reaction. This research conceivably can establish the basis of a method for manufacturing $SiC-Si_3N_4$ composite powder using a CO_2 laser.

Application Field

This research showed how SiC fine particles can be synthesized for use as a sintering raw material for new ceramics and was performed as the basic research for $SiC-Si_3N_4$ composite fine particle manufacturing. This technique is also applicable to the manufacturing of other ceramic-related substances.

Functionally Gradient Material Generated by Self-Propagating High Temperature Synthesis

430672151 Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 266-267

[Article by Government Industrial Research Institute, Tohoku]

[Text] **Summary**

The new material R&D referred to as functionally gradient material (FGM) development is being promoted as a science and technology promotion adjustment theme "Research on Basic Techniques for Functionally Gradient Material Development for Thermal Stress Relaxation." The thermal stress relaxation-type functionally gradient material as a superheat-resistant material, the development target, is expected as one of the key technologies in the development of the materials for the second-generation space shuttles and nuclear fusion. Functionally gradient materials, which function in a sharp temperature-gradient potential field, are created according to the concept by which the conventional way of thinking has been reversed and made to jump, i.e., these materials are aimed at minimizing the thermal stress that occurs under a sharp temperature grade by unevenly synthesizing a material so that its composition is designed in advance.

As methods for synthesizing FGM, the physical or chemical evaporation method, powder metallurgy method, melt-injection method, and self-propagating high temperature synthesis method (SHS) are the main ones that have been studied, but this article briefly discusses the SHS being studied at the Government Industrial Research Institute, Tohoku. The functionally gradient material created by the self-propagating high temperature synthesis method is termed SHS-FGM in this article.

Application Fields

Research on FGM is being promoted for use in the airframe and engine interior wall materials of the Japanese space shuttle, but FGM will also serve as an effective material in other fields. If heat-resistant and heat-shielding properties are taken as examples, there is a large possibility that FGM will replace the material forming the interior walls of nuclear fusion reactors, liner materials of various engines, and conventional metal-ceramic joining

materials. Also, if research is initiated not only on thermal or mechanical gradient materials, but also on electric, chemical, optical, nuclear and biological functionally gradient functions, the application fields will extend infinitely.

Detailed Contents

1. Self-Propagating High Temperature Synthesis Method (SHS)

This method was developed in the Soviet Union and is a method of synthesizing ceramics and intermetal compounds. It is generally called SHS (self-propagating high temperature synthesis), but in Japan it is referred to by such names as self-exothermic, self-endothermic, self-propagating, or combustion synthesis, depending on the characteristics of the synthesis process. The characteristics of SHS are briefly described here. As shown in Figure 1, this synthesis is characterized by the igniting of a part of the raw material powder, and the spontaneous propagation of the synthesis reaction from the ignited part. Therefore, this method offers various advantages, such as not requiring heat to be provided externally, synthesis advances within several seconds, it can obtain a very high temperature, and is energy and cost saving. This is a field in which research has been rapidly initiated during the past few years.

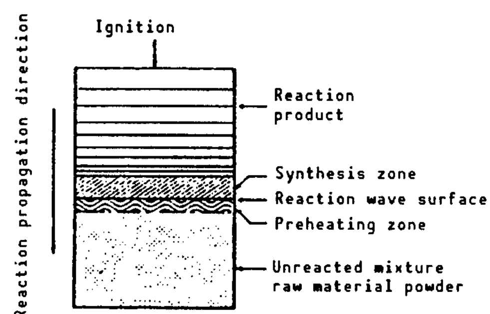

Figure 1. Principle of Self-Exothermic Reaction Method

2. SHS-FGM

Figure 2 shows the basic design concept of SHS-FGM. A single layer of ceramics is used on the high-temperature side which requires heat resistance, and a composite layer consisting of ceramics and a metal whose composition has been made gradient is installed between the high-temperature layer and the metal used on the cooling side. The basic structure of SHS-FGM involves unified synthesis and the formation of these ceramic and composite layers on the basic metal. Figure 3 shows the flow of FGM development with the self-exothermic reaction method.

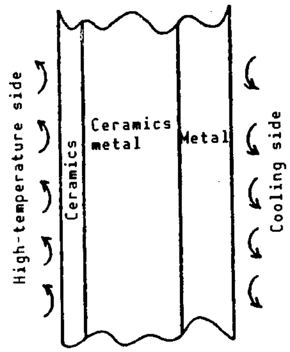

Figure 2. Concept of SHS–FGM Basic Design

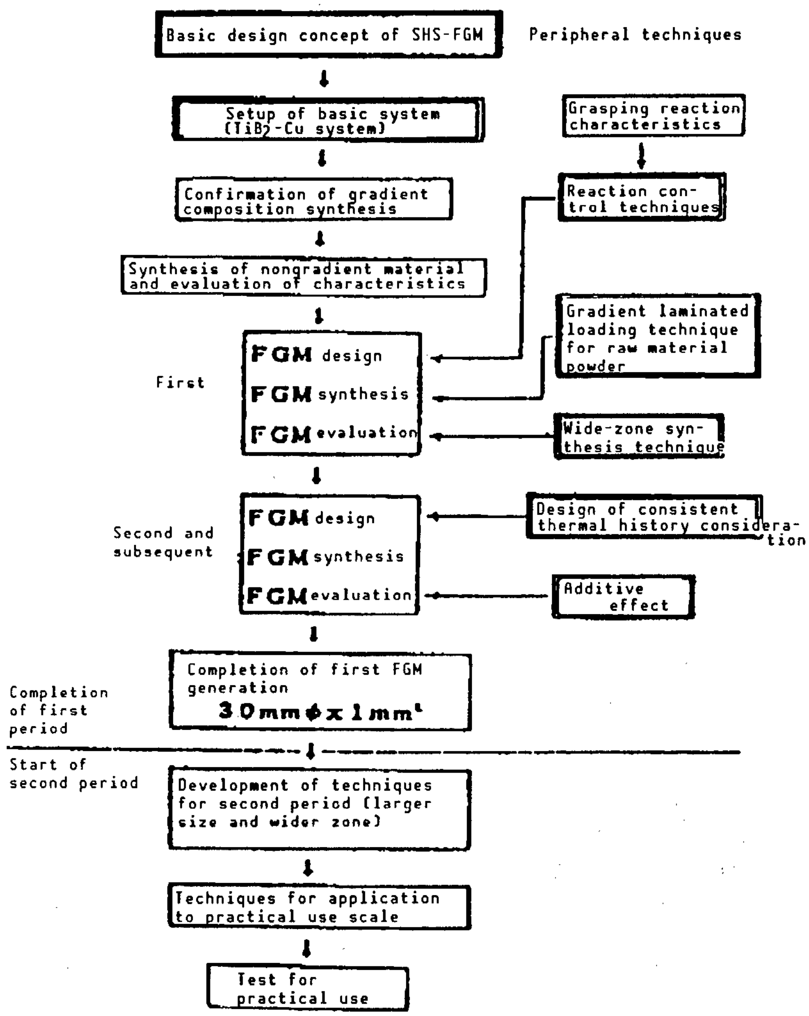

Figure 3. Flowchart for FGM R&D by Self-Exothermic Reaction Method

3. Material Design

The material design for obtaining FGM is as follows. The gradient component distribution for minimizing thermal stress is obtained from the thermal and mechanical characteristics of the TiB_2•Cu system. In the basic design, as shown in Figure 2, the surface consists of a single layer of TiB_2, and the material design progresses so that the Cu component distribution is determined from the area where the temperature distribution inside the material does not exceed the melting point of Cu. Figure 4 shows the component distribution obtained for the TiB_2•Cu system by numerical calculation. The area in which x=0.0 to 0.1 is the part of the surface where TiB_2 is 100 percent, the area in which x=0.1 to 1.0 is the intermediate layer where TiB_2 gradually decreases and Cu gradually increases, and the area where x=1.0 is the basic body surface where Cu is 100 percent. The results in Figure 4 are based on the infinite plane model, and results of applying this technique to the infinite cylinder model have also been obtained.

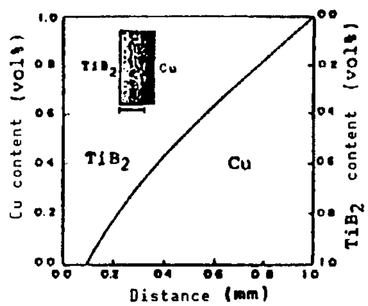

Figure 4. Design of TiB_2–Cu System FGM (infinite plane model)

4. Structure Control

A description of the TiB_2–Cu system FGM synthesis is as follows. Since Cu has not formed boride at the time of TiB_2 synthesis, it takes on the TiB_2–Cu (or Cu alloy) system two-phase mixture structure. Since an arbitrary compound can be introduced into a reaction system with the TiB_2–Cu system as the basic component, the thermal, mechanical, and atmosphere-resistant characteristics can be adjusted as required by adding carbide, nitride, or oxide powder during synthesis. Also, if too many metals do not participate in the reaction during the ceramic-metal system synthesis, the reaction comes to a stop, but in FGM, the heat quantity necessary for reaction and formation is supplemented in the form of supplying the reaction heat of the strong reaction side to the metal-rich side where the reaction heat is low, thereby using the reverse process of obtaining an uneven composition structure. This method allows a material to be acquired which is of a continuous phase, ranging from the single TiB_2 phase to the phase in which Cu accounts for 100 percent.

The characteristic of the material manufacturing method using the self-propagating high temperature synthesis method consists of the simultaneous synthesis and formation technique. Such techniques have already been proposed, but Government Industrial Research Institute, Tohoku, is examining a method to perform synthesis and formation under static hydraulic pressure as its SHS-FGM technique.

5. Evaluation of Characteristics

The characteristics of SHS-FGM have not been evaluated yet, but the test equipment is being installed to test heat shielding, thermal fatigue, and thermal shock.

Notes, Etc.

Creation of functionally gradient materials by the self-propagating exothermic reaction method involves many subjects requiring examination, such as grasping reaction characteristics (reaction control technique), the low-pressure and wide-range synthesis technique, gradient laminated filling technique for raw material powder, composition corresponding to the actual environment (additive control), and design consistently taking thermal history into consideration. However, the effect of these development techniques is expected to extend very far. FGM is positioned not only as functional coating, but also as a new composite material. Therefore, the authors would like to suggest that the word "complexing" be used particularly for the synthesis of such materials as FGM.

Ceramic Coating Method for Curved Pipe Interior by Reaction Melting, Attaching Method

43067215m Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 268-269

[Article by Government Industrial Research Institute, Tohoku]

[Text] **Outline of Target Enterprise**

Name of enterprise:	Kurimoto Iron Works, Ltd.
Number of employees:	3,428
Capital fund:	¥9.1 billion
Sales amount:	¥102.7 billion
Kind of business:	Cast iron pipe and iron truss manufacturer

Situations for Which Guidance Is To Be Provided

The thick wall ceramic coating technique (reaction melting and adhesion method) developed by the Government Industrial Research Institute, Tohoku, uses the thermite reaction, and performs ceramic coating by continuously solidifying and sticking the melted ceramics created simultaneously with the reaction on a metal surface, such as the interior surface of an iron pipe, as the reaction advances. Use of the reaction melting and adhesion method has facilitated the formation of thick wall ceramics, consisting mainly of alumina, on the inside surfaces of pipes of varying shapes such as bent pipes. Since the composite pipe on which the coating was applied by the reaction melting and adhesion method is expected to have superior characteristics in corrosion resistance, abrasion resistance, thermal resistance and adiabatic properties, Kurimoto Iron Works, Ltd., requested guidance from this institute, and, in response, was provided with technical guidance, particularly regarding the technique for thick wall ceramic coating on the inside surface of a bent pipe using the reaction melting and adhesion method.

Guidance Contents

The representative thermite reaction is shown by the following formula:

$$2Al + Fe_2O_3 = Al_2O_3 + 2Fe + 856KJ$$

The temperature (highest reaching temperature) of the reaction system reaches a temperature as high as about 3,700 K, and high melting point alumina (melting point: 2,324 K), as well as metallic iron, enter melted states. During the actual coating, the highest reaching temperature is adjusted to approximately 3,400 K through the addition of additives.

Figure 1 shows the principle of the reaction melting and adhesion method. First, a thermite agent fills the inside of the metal pipe by pressing, and the upper metal wall is kept almost vertical. The reaction advances continuously and the melted ceramic generated is held in the form of a pool at the upper part of the reaction system. The metallic iron sinks due to the difference in specific gravity, the melted ceramic cools and solidifies at the part joined to the metal wall, and becomes joined to the metal wall. The reaction advances at a speed of about 10 mm/s, and the ceramic coating progresses continuously as the boundary surface of the reaction drops.

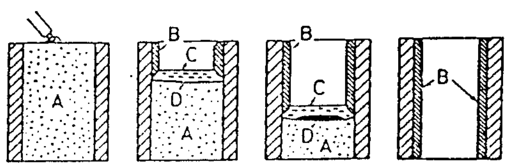

Figure 1. Basic Principle of Reaction Melting and Adhesion (A: Thermite agent; B: Ceramic layer; C: Melted ceramics; D: melted metal)

As part of the technical guidance, the basic research in the technique for coating a ceramic to the inside surface of the straight pipe was first performed with a thermite reaction, and the application limitations when coating using the reaction melting and adhesion method, that is, the internal diameter, length, and wall thickness of the pipe, the iron oxide and aluminum to be used as the thermite gent, and various additives, were examined. Also, since uneven coating occurred at the upper and lower ends of the pipe, an approximately 30 mm long paper dummy was connected to obtain good coating ends.

When the reaction melting and adhesion method is applied to the ceramic coating of a curved pipe, the curved pipe must be rotated slowly in its circumferential direction as the thermite reaction advances at an appropriate speed, as shown in Figure 2. Therefore, the authors manufactured an experimental device which allows an appropriate rotation speed to be set up and performed coating experiments on the internal surface of curved pipes. When coating is performed on the internal surface of bent pipe, the boundary surface of the reaction inevitably becomes inclined to the side of the internal circumference as the boundary surface of the reaction drops due to the difference between the internal and external circumferential lengths. If 1) the length of the bent pipe is short, 2) the pipe diameter is small or 3) the radius of curvature is small, the influence exerted by the inclination of the boundary surface of the reaction is small and negligible, but if these values become larger, good coating becomes difficult to conduct by the uniform

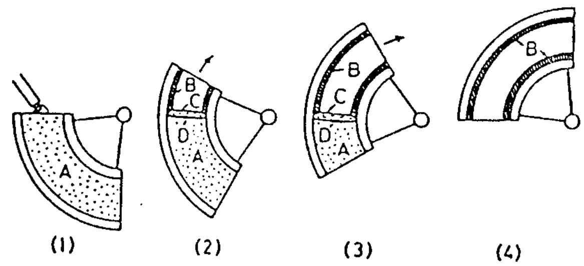

Figure 2. Application of Reaction Melting and Adhesion Method to Bent Pipe (A: thermite agent; B: Ceramic Layer; C: Melted ceramics; and D: Melted metal)

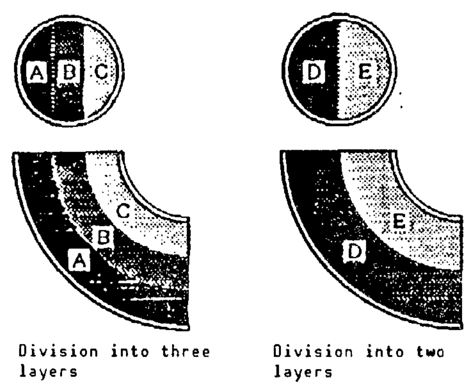

Figure 3. Nonuniform Loading Method of Thermite Agent (Division into three and two layers)

filing of the thermite agent alone. As a means of resolving this problem, the method of filling thermite agents of different reaction propagation speeds in the direction from the inner circumference to the outer circumference of the curved pipe (uneven filling) is used. A continuously adjustable reaction propagation speed is ideal, but is practically impossible to achieve. Therefore, a three- or two-layer structure (Figure 3) has been adopted. By using the reaction propagation speed data shown in Figure 4, thermite agents were selected and filled so that the reaction propagation speeds on the individual center circumferences became faster on the outer circumferences. In this way, coating experiments could be conducted under the conditions that the boundary surface of the reaction remained in a horizontal position in a rotating curved pipe and did not incline to the inner circumference side, and good coating could be applied to the internal surface of a curved pipe with a large curvature. Figure 5 [not reproduced] shows a sample of the bent pipes to which coating has been applied successfully.

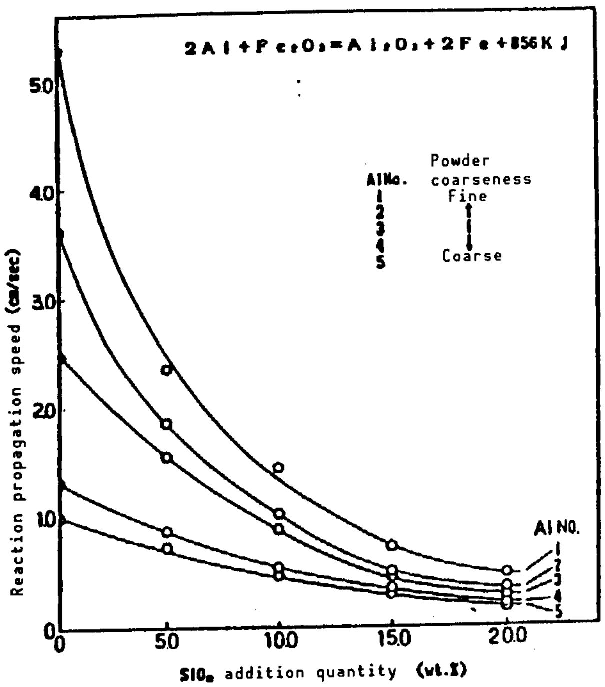

Figure 4. Influence of Granularity of AI and SiO_2 Addition Quantity on Reaction Propagation Speed

Technical Results and Effects

Based on the above-mentioned research results, coating experiments were performed with importance attached to 90° long elbow pipes (2-inch pipe, 53-mm inner diameter), and research for improvement is continuing to be performed.

The subjects for examination are testing and evaluation of corrosion resistance and abrasion resistance involving a bent pipe, coating experiments on flange pipes, enhancement of the smoothness of ceramic surfaces, and reduction of the iron oxide which exists in ceramics and exerts a bad influence on corrosion resistance. Research and technical guidance are currently being promoted to resolve these topics.

Additional Information

Related patent: Patent applied for 62-251140.

Diffusion Joining of Ceramics

43067215n Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 270-271

[Article by Government Industrial Research Institute, Nagoya]

[Text] **Summary**

This research is aimed at establishing a technique to join two ceramic pieces. The authors thought that if high-temperature diffusion joining were used, a high joining strength able to endure use at high temperature could be expected, and forge welding was conducted by means of a hot press. The sample material was a silicon nitride ceramic, the influence of the joining temperature (1,400~1,600°C), pressure (to 40 MPa), holding time (0.5~10 hours), and roughness of the joining surfaces (0.7~1.2 μm) on the joining strength was examined, and appropriate conditions were investigated. In addition, in joining different materials, the thermal stress caused by the difference between the coefficients of thermal expansion of the individual materials sometimes caused cracks to occur in the joined part. Therefore, how the failure probability of joining material changes depending on the dispersion of member dimensions and strength was studied from the thermal stress distribution calculated by the finite element method and the rupture stress distribution of ceramics obtained by Wible statistics. The results of the joining experiments and stress analysis are summarized as follows: 1) to raise the joining strength, smoother joining surfaces and higher joining temperature and pressure are preferable under the above-mentioned conditions. At this time, there is no need to lengthen the joining time; 2) the failure probability of the joining part is remarkably influenced by the member diameter. The larger the diameter, the more likely failure will occur. It has been found that if the Wible coefficient of the member is small at this time, the failure rate increases further.

Detailed Content

The ceramic used in the joining experiment is hot-pressed silicon nitride, its bending strength averages 1 GPa, and its Wible coefficient is 8.2. This material contains 2.9 percent alumina and 5 percent yttria as the additives at sintering. The testpiece for joining is a rectangular prism, 15 x 15 x 19 mm, and its surface roughness has been adjusted by diamond grinding. In joining,

polyethylene was used as the adhesive, and the testpieces were fixed by melting polyethylene in a vacuum and at 170°C. The fixed testpieces were then set in a graphite mold and high-temperature joining was performed by means of a hot press. To evaluate the strength of the joining material, a bending testpiece (3 t x 4 w x 30 l mm) was cut out from the joining material and was subject to a four-point bending test. Figures 1 and 2 show the influence of the joining temperature and pressure on the joining strength, and indicate that the higher temperature and pressure yield stronger joining and that the polyethylene used as the adhesive during preprocessing effectively enhances the joining strength. The effect of temperature and pressure is thought to greatly contribute to the increase in strength by gradually deforming the materials near the boundary surface of the joining and increasing the true contact area. Since this material deformation occurs creepwise, the influence of the holding time at high temperatures must be examined in advance.
Figure 3 shows the results. Even if the holding time is lengthened under low temperature and pressure conditions, the joining strength does not increase. The authors suggest that under high temperature and high pressure, the creep deformation speed increases, the contact surface increases with the passage of time, and the joining strength increases. This tendency is noticed, but the joining part does not need to be held for a long time.

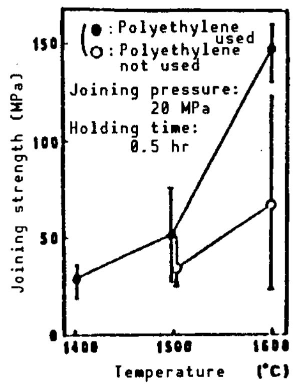

Figure 1. Influence of Joining Temperature on Joining Strength

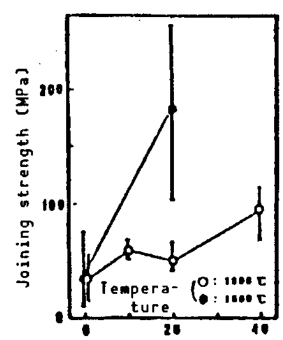

Figure 2. Influence of Joining Surface Pressure on Joining Strength (Holding time: 1 h)

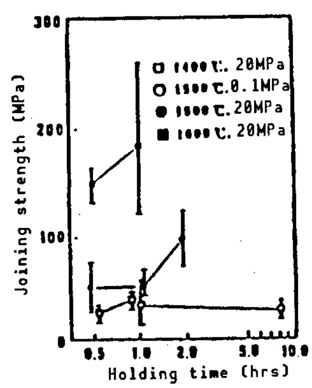

Figure 3. Influence of Holding Time on Joining Strength

If the joining strength is thought to change with the contact area, it is anticipated that it will be influenced by the roughness of the joining surface. Figure 4 shows the relationship between the roughness of the joining surface and the joining strength. The black symbols in the figure show the joining performed under the temperature and pressure that promote creep deformation. Under such conditions, the joining strength is found to be sharply enhanced by decreasing the surface roughness (0.22 μm or less for this material). This means that if the viewpoint is changed, strong joining will be difficult to obtain when the evenness and roughness of the joining surfaces are bad, even if the temperature and pressure are raised.

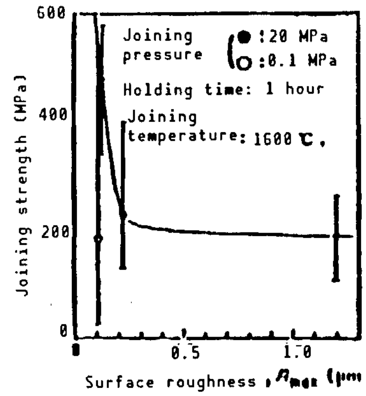

Figure 4. Influence of Surface Roughness on Joining Strength

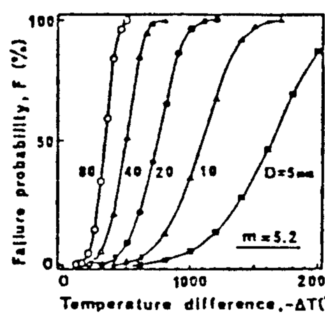

Figure 5. Influence of Member Diameter on Failure Probability of Joining Agent of Si_3 and SiC

When materials with different coefficients of thermal expansion are used, the cracks caused by thermal stress at the joint pose a problem. Figure 5 is a sample calculation showing the influence of the joining material dimensions on the failure probability of the joint, and assumes joining between silicon carbide and silicon nitride. If the member diameter increases, the failure rate becomes higher, even if the joined shape is similar and the stress is the same. Now, it is thought that if the strength of the ceramic itself is taken into consideration, the defect rate will increase as the material becomes larger, the strength becomes smaller and the strength dispersion becomes greater. By means of a sample calculation, Figure 6 shows how the failure probability of the joined part changes depending on the dispersion of the

strength of the ceramic itself. Even if the average strength is the same, failure occurrence is facilitated when the strength dispersion is large (Wible coefficient m value is small). In other words, a large temperature difference cannot be endured. To make the joining of different ceramic materials possible, it is indispensable that the average strength of the material itself be raised and the strength dispersion be decreased, and close coordination of the manufacturing and processing techniques becomes increasingly necessary.

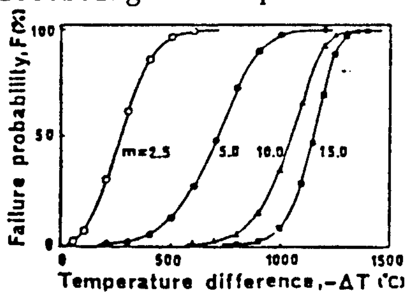

Figure 6. Variation of Failure probability With Wible Coefficient m of Member (SiC) (Member diameter = 20 mm, thickness = 10 mm)

Performance

(1) Joining of the same kinds of ceramics

The joining temperature, pressure and holding time are determined in the range where creep deformation occurs on the boundary surface of the joining and the contact area increases. When two silicon nitride materials were joined, an approximate joining strength of 200 MPa was obtained under the conditions of 1,600°C, 20 MPa, and holding time of 1 hour. If, at this time, the surface roughness of the joining surfaces is less than about 0.2 μm, the joining strength can be raised. Also, the joining strength becomes higher when polyethylene is used as an assistant joining agent.

(2) Joining of different kinds of ceramics

With the thermal stress that occurs due to difference between coefficients of thermal expansion, cracks occur near the joined part. The authors analyzed the failure rate of the joined part by calculating the thermal stress distribution and material strength dispersion, and confirmed the influence of the joined part dimensions and material strength characteristics on the failure probability. To make the joining of different ceramics possible, it is indispensable that the average strength be raised and dispersion minimized simultaneously.

Application Fields

These include fields related to automobiles and energy, such as engines and turbines, which require strength and abrasion resistance at high temperatures, and production processing fields which utilize forming molds.

High-Temperature Oxide Superconductive Material Manufacturing, Its Electromagnetic Properties

43067215o Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 276–277

[Article by Government Industrial Research Institute, Nagoya]

[Text] **Summary**

The authors manufactured submicron particles of Y–Ba–Cu–O and Y–Ba–Cu–(F, O) system oxides, with critical temperatures at the 90 K level, using the coprecipitation method, and obtained a sintered substance by pressing and sintering the powder. They investigated the superconductivity of the obtained sintered substance by measuring its specific resistance, magnetic susceptibility, and Meissner effect. In powder synthesis, particles with diameters of from 0.2~0.5 μm were obtained at a distribution rate 80 percent or more, and the volume ratio of the superconductive phase was 80 percent or more. The critical temperature of the sintered substance was 90~97 K and its diamagnetic susceptibility was -10^{-3}emu/g at 77K (liquid nitrogen temperature). The superconductivity was found to improve considerably with low-temperature annealing in a current of air and oxygen.

Detailed Contents

(1) Powder synthesis

a) Nitrates of Y, Ba, and Cu were dissolved in distilled water so that the designated component ratio was attained by adjusting the pH, dripping the solution into oxalic acid solution using a burette, and causing an oxalate to be created. The remaining unprecipitated ions (mainly Cu^{2+} ions) were precipitated by using hydrazine. The solution stirred during this procedure.

The precipitated substance was dehydrated and dried by the freezing and drying method. This dried powder was crushed and mixed in a mortar and heated in an alumina crucible with an electric furnace in air at 600°C for 2 hours to obtain a Y–Ba–Cu–O system oxide powder by decomposing the oxalate.

b) To synthesize the Y–Ba–Cu–(F, O) system oxide, a portion of the Y^{3+} and Ba^{2+} ions were first precipitated as fluoride by dripping the calculated quantity

of HF into the solution containing Y^{3+}, Ba^{2+} and Cu^{2+} ions and using the same method as the Y-Ba-Cu-O system oxide synthesis method.

A compound having a specific crystal structure (tetragonal crystal and prismatic crystal) was obtained by further heat-treating the temporary sintered powder obtained by the method mentioned in a) and b) at 800~950°C.

The $YBa_2Cu_3O_{7-\alpha}$ powder synthesized by this method has particles of diameter 0.2~0.5 μm, its distribution rate is 80 percent or more, and 80 percent or more of the particles have been found to be superconductive substances by measuring their diamagnetic susceptibility. Figure 1 shows an SEM photograph and X-ray diffraction diagram of the $YBa_2Cu_3O_{7-\sigma}$ powder. This figure clearly

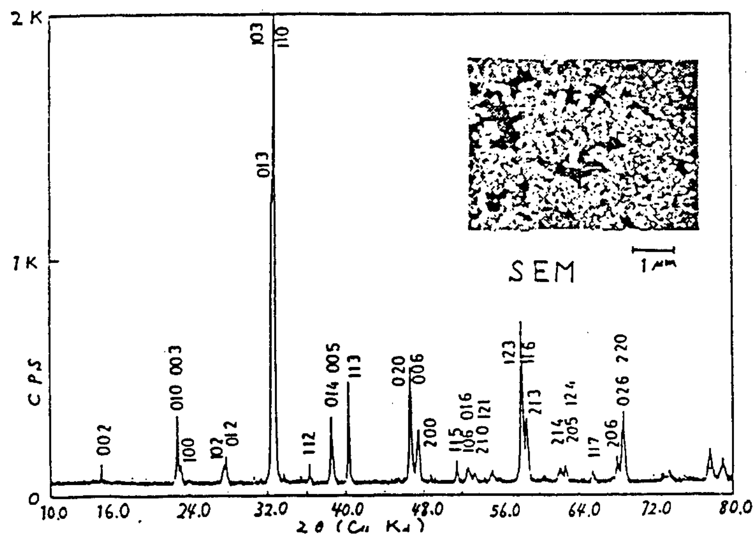

Figure 1. X-Ray Diffraction Profile of $YBa_2Cu_3O_{7-\sigma}$ Powder
(Heat treatment: 900°C, 4 h, in air)

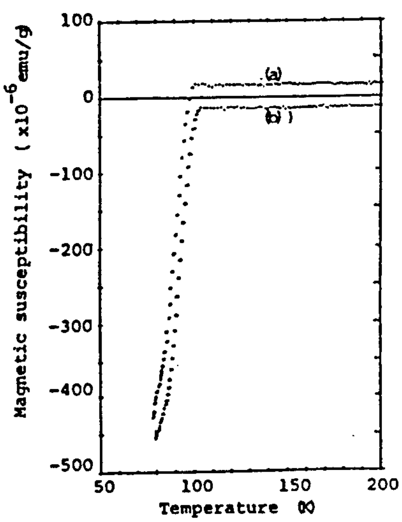

Figure 2. Magnetic Susceptibility of $YBa_2Cu_3O_{7-\sigma}$(a) and $YBa_2Cu_3(F,O)_{7-\sigma}$(b)

shows that the powder consists mostly of prismatic crystals. Figure 2 shows the measurement results of the magnetic susceptibility of $Yba_2Cu_3O_{7-\sigma}$ and $YBa_2Cu_3(F, O)_{7-\sigma}$. Tc is higher by about 2 K when doped with fluorine.

(2) Manufacturing of Sintered Substance

A sintered substance was manufactured by press-forming the powder synthesized by the method mentioned in (1) and sintering the formed substance in an electric furnace using platina foil or an alumina plate at 900~950°C for 4~46 hours. The sintered substance was then annealed in a flow of air or oxygen in a cylindrical furnace at 300°C for 30 hours. The results of X-ray diffraction and a magnetic susceptibility measurement showed that the volume factor of the superconductive phase (prismatic crystal) was 70 percent or more after sintering and annealing.

(3) Electric Resistivity of Sintered Substance

The sample was fixed by the pressed fixing method and its electric resistance was measured at from room temperature to liquid nitrogen temperature by the direct current four-terminal method. Figure 3 shows the electric resistance measurement results for the $YBa_2Cu_3O_{7-\sigma}$ sintered substance. The resistivity did not become zero immediately following sintering, and the substance entered the superconductive state after several times of annealing, with Tc = 95 K (off set) and ~97 K (on set).

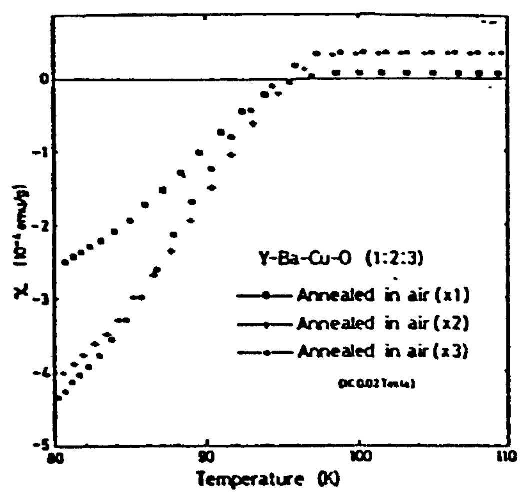

Figure 3. Electric Resistance of $YBa_2Cu_3O_{7-\sigma}$
(x i: heat treatment at 300°C and for 10 hours)

(4) Magnetic Susceptibility of Sintered Substance

To measure the magnetic susceptibility, the Faraday method using a torsion balance was used. The measurement temperature range extended from room temperature to the liquid nitrogen temperature. Figure 4 shows the magnetic susceptibility measurement results for the $YBa_2Cu_3O_{7-\sigma}$ sintered substance. Like the variation of electric resistivity, the diamagnetic susceptibility

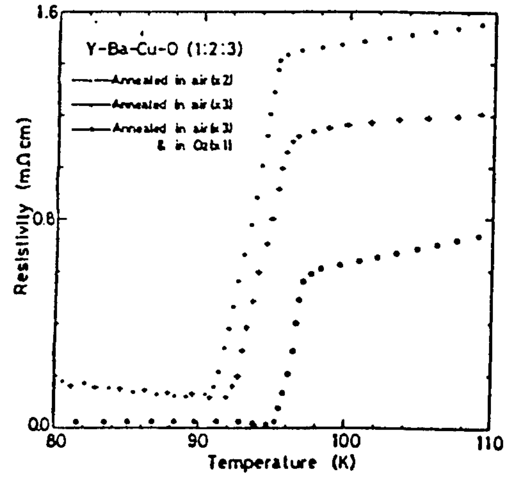

Figure 4. Magnetic Susceptibility of $YBa_{2XCu}3O_{7-\sigma}(\chi)$
(x i: heat treatment at 300°C and for 10 hours)

increased immediately following sintering as the number of annealing treatments increased and χ80 K = -25×10^{-4}emu/g. In addition, as a result of examining the Meissner effect at 77 K, $\chi = -1.2 \times 10^{-3}$emu/g.

Performance

The synthesized powder had a particle diameter of from 0.2~0.5 μm with an 80 percent distribution rate, and consisted of a superconductive phase (prismatic crystal $YBa_2Cu_3O_{7-\sigma}$) of 80 percent or more. For the sintered substance, Tc = 95 K (off set) to 97 K (on set) and $\chi = -4.4 \times 10^{-3}$emu/g. The volume factor of the superconductive phase was 70 percent or more.

Application Fields

Superconductive electronic devices, magnets, power transmission, and power storage (in R&D stages and prediction).

Patent

Patent application scheduled.

Manufacturing of Oxide Optical Semiconductor

43067215p Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 286-287

[Article by Government Industrial Research Institute, Nagoya]

[Text] **Summary**

A new material development technique which is attracting attention is the so-called melted substance super-rapid cooling method (super-rapid cooling method) which melts a material and rapidly cools and solidifies the melted substance to a thin film by means of a roller rotating at high speed. When various ceramic thin films were manufactured by using this super-rapid cooling method and their characteristics were examined, the Bi_2O_3 system $PbO-TiO_2$ system super-rapidly cooled films were found to show considerably high optical superconductivity. The cause of this optical superconductivity is thought to be that the structural defects generated at high temperatures were frozen to room temperature and formed an electron structure that facilitated the generation of optical superconductivity. The optically superconductive film obtained is faulty in that its resistance value is high, but since its responsiveness for visible light is extremely high, and since a film whose resistance value in the optically excited state differs from that in the dark state by 10^2 can be easily repeated, it is expected to be usable as an optical switch element and light-detecting material.

Detailed Contents

Super-rapid cooling materializes amorphous materials, oriented crystals, and transparent materials, depending on the kind of material, freezes high-temperature of quasi-stable phases, and is usable as a measure to forcibly increase the solid solution of a different ion. Also, since these reactions occur in a very unbalanced state, a frozen structure contains many defects. Therefore, the use of the super-rapid cooling method allows materials to be synthesized which have structures and forms unattainable by conventional techniques and it is expected to lead to the enhancement of material characteristics and the appearance of new functions.

This institute has been conducting research on the synthesis of ceramic thin films by the super-rapid cooling method and evaluating their characteristics,

and has found several new interesting facts. The oxide optical-semiconductor introduced in this article is one of them.

Additives for Bi_2O_3 include Y_2O_3, TiO_2, Nb_2O_5, MoO_3, and WO_3, with the quantity of the additive ranging from 0~15 mol percent.

TiO_2 and N_2O_5 were used for PbO, and were added in the range of from 0~60 mol percent. The mixture of the designated composition was first finished to a sintered stick of about 5 x 5 x 30 mm^3 by a procedure involving tentative burning, remixing, forming and sintering. The tentative burning and sintering were performed at 700~800°C for 5 hours in air. The sintered stick was super-rapidly cooled and formed into a thin film by a biroller device provided with an infrared ray converging heating furnace.

Bi_2O_3 has various shapes, such as α (simple prismatic), β (tetragonal), γ(bcc), and δ(fcc), but if it is super-rapidly cooled with a second component added, the β, β^-, and δ phases can be successively separated, independent of the second component. The β^- phase here is a tetragonal quasistable phase which is the β phase whose order structure has been disordered. Figure 1 shows the relationship between the lattice constants and compositions of the β, β^-, and δ phases generated in super-rapidly cooled films of Y_2O_3, TiO_2 and Nb_2O_5 as representative examples. The figure shows that the lattice constant change of the β and β^- phases does not follow the Vegard's Law. Since Y_3^-, Ti^{4-} and Nb^{5+} are smaller than Bi^{3-}, the volume should decrease as their addition quantity increases, but it is recognized that when Y_2O_3 or Nb_2O_5 is added, the volume gradually increases, while when TiO_2 is added, the volume does not change noticeably. Also, since $Vo^{1/3}$ is lower than the value extrapolated from α in the δ phase, the β and β^- structures are found to be more dense than the δ structure. As for the reason for such a contradicting phenomenon occurring, it may be worth investigating whether or not the super-rapid cooled phase takes the normal positive ion distribution. That is, even if the second component is added, some of the positive ions enter positions other than the regular lattice positions (for example, (1/2, 1/2, 1/2)), the lattice shrinkage corresponding to the addition quantity is small, and a dense structure materializes.

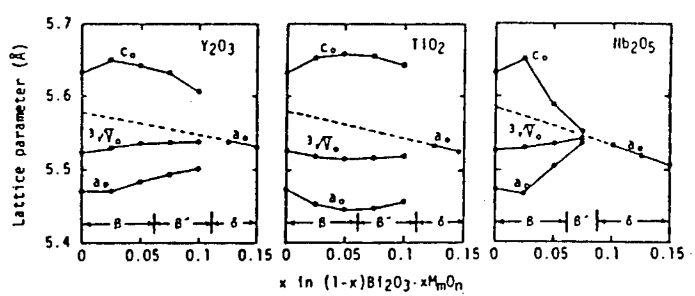

Figure 1. Relationship of Lattice Constants and Compositions of β, β^- and δ Phases Generated on Bi_2O_3-Based Super-Rapidly Cooled Films

One of the noteworthy characteristics of the Bi_2O_3 system super-rapidly cooled film is that the film exhibited high optical superconductivity. To enhance the optical conductivity of this super-rapidly cooled film, the addition of the second component is necessary, and not only many oxides but also fluorides, such as CaF_2, YF_2 and BiF_3, have been confirmed as being effective components. The optimum addition quantity depends on the kind of additive, but ranged from 5 ~10 mol percent. This range corresponds to that in which the β and β^- phases are generated. Figure 2 shows the relationship between the surface resistivity and the wavelength of the irradiated ray of the Bi_2O_3–MO_3 (M = Mo, W) system super-rapidly cooled film as examples of films exhibiting comparatively high optical superconductivity. In the figure, the horizontal line shows the resistance value in the dark state. The Bi_2O_3 system super-rapidly cooled film is characterized by a remarkable resistance value reduction shown when a ray (based on blue and green) near 500 nm is irradiated.

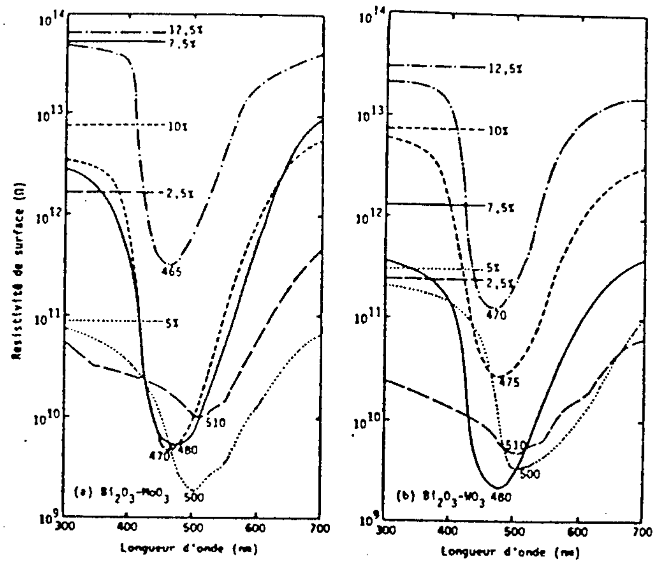

Figure 2. Relationship of Surface Specific Resistance and Wavelength of Irradiated Light of Bi_2O_3–MO_3(M+Mo,W) System Super-Rapidly Cooled Film
(The percent in figure shows added quantity of MO_3.)

In general, the substance causes defect centers that deviate from the stoichiometrical composition and thermal excitation, and electrons and positive holes are trapped in levels in the forbidden band. When these electrons and positive holes are given optical energy and excited to the conductive band, the optical conduction phenomenon occurs. In the cases of the super-rapidly cooled film as well, the structural defects in the equilibrium state at high temperatures are thought to be frozen to room temperature and form an electron structure facilitating electron conduction. In the Bi_2O_3–M_2O_5(M = V, Nb, T) system, optical superconductivity appeared only in the super-rapidly cooled film which had generated the β and β^- phases, but in the Bi_2O_3–MO_3 system, optical superconductivity was also observed with a thin film consisting of the δ phase. From this fact, the defect state rather than the film structure seems to be the important factor in bringing about optical superconductivity to a super-rapidly cooled film.

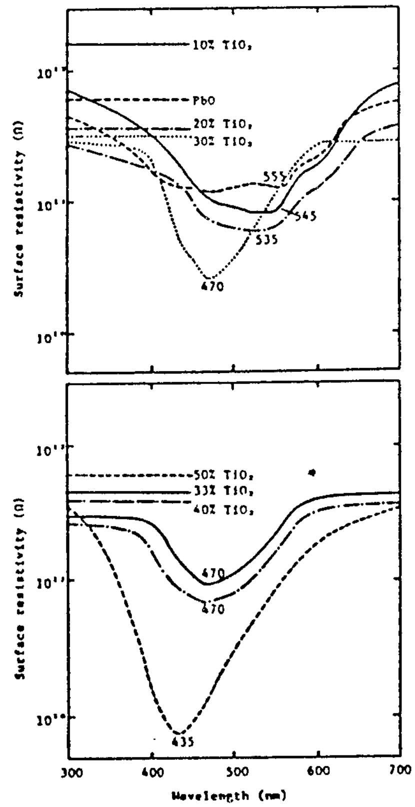

Figure 3. Relationship of Surface Specific Resistance and Wavelength of Irradiated Light of $PbO-TiO_2$ System Super-Rapidly Cooled Film

With the PbO system, the TiO_2-added super-rapidly cooled film demonstrates comparatively high optical superconductivity, while the Nb_2O_3-added one exhibits only slight optical superconductivity. Figure 3 shows the optical superconductivity curves of $PbO-TiO_2$ system super-rapidly cooled films. This figure shows that a tetragonal PbO_2 solid solution is generated when the added quantity of PbO is 10~30 percent, a PbO solid solution and $PbTiO_2$ are generated when the quantity is 33~40 percent, and $PbTiO_2$ is generated when the quantity is 50 percent. Optical superconductivity is found to be higher in the solid solution containing TiO_2 than in pure PbO_2. It should also be noted that $PbTiO_2$, recognized as a ferromagnetic substance, exhibits the strongest optical superconductivity. As clearly shown by the fact that TiO_2 is an effective material for the semiconductor electrode used in the optical decomposition of water, TiO_2 belongs to the optically conductive group. TiO_2 is thought to promote the optical superconductivity of PbO, rather than obstruct it, because of the optical properties of TiO_2. A comparison of the lattice constants of the $PbTiO_3$ obtained by super-rapid cooling and that obtained by the usual method showed that the former has smaller tetragonal anisotropy and takes a more dense structure. In this case, it is also presumed that an abnormality occurred in the positive ion distribution, and that this abnormality caused the optical superconductivity.

Performance

Since this optical semiconductor is obtained in the form of a thin film, it has the advantage that it is usable immediately as an optical element if an electrode is attached to it. Its weak point is that the operating voltage must be made higher because the resistance value of the thin film is quite high. However, this problem can be settled by using a comb-like or transparent electrode and making use of the resistance value in the direction of the film thickness.

Application Field

Optical switch elements or light-detecting materials.

Notes, Etc.

None.

Others

Patent 1987-4678732 "Manufacturing method for optical semiconductor"

U.S. Patent (1987) No 4,678,732 "Manufacturing method for optical semiconductor"

Superconductive Thin Film Material Technique

43067215q Tokyo SENTAN KOGYO GIJUTSU OYO YORAN in Japanese Mar 89 pp 477-478

[Article by Electrotechnical Laboratory]

[Text] **Summary**

Oxide-based high-temperature superconductors applicable at the liquid nitrogen temperature are all composite oxides consisting of elements whose vapor pressures and degrees of chemical activity differ greatly. Therefore, in order to make thin films of these materials and apply these films to the electronics field, a thin film synthesis technique that can strictly control film composition and the degree of oxidation is important. In this project, a process for synthesizing the oxide superconductor thin film was investigated from the viewpoint of developing a film growth element process control method, the mechanism of the film composition fluctuation in the sputtering method, which is the main current of the oxide superconductor film synthesis method, was explained, and a composition control method was developed based on the mechanism. This method controls the incidence of high-speed electrons on a growth surface, which is the main cause of composition fluctuation, and is capable of synthesizing a high-quality oxide superconductor thin film in the as-grown state.

Detailed Contents

1. Synthesis Method

From research on compositional changes of the Sr-La-Cu-O and Ba-Y-Cu-O system films, the main cause of film composition changes has been found to be that relatively weak bonds in the structure, such as Ba-O and Cu-O, are weakened or cut by the incidence of high-speed electrons released from the sputtering target on the growth surface and that selective deficiency of these elements occurs. Therefore, to materialize superior composition control in sputtering synthesis of oxide superconductor thin films, this electron impact must be suppressed.

In this research, the authors have developed the method of applying an electron deflecting magnetic field, as shown in Figure 1(a), and performing

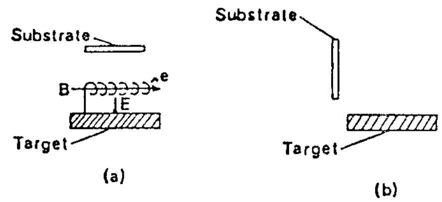

Figure 1. Method of Suppressing Incidence of High-Speed Electrons on Growth Surface
(1) Method applying electron deflecting magnetic field
(2) Method based on layout of target and substrate

film growth in a space where no high-speed electron exists, as shown in Figure 1(b). The former suppresses the incidence of high-speed electrons on the growth surface with a magnetic field of several hundred gauss, and is easily applicable to a general device in which the substrate and target are facing each other. The latter uses the phenomenon that, since the electrons are accelerated by the electric field applied to the target, the high-speed electron density drops suddenly outside the space formed by translating the target in the opposite direction of the electric vector.

2. Structure and Characteristics of Synthesized Film

To check these methods, the authors formed thin films using an oxide superconductor of stoichiometric composition as the target. Table 1 shows the relationship between the composition and sputtering gas pressure of the film formed by the method shown in Figure 1(a) (electron deflecting magnetic field: 150 gauss), with $Ba_2YCu_3O_y$ at the 90 degree level as the target. In this case, a film with the same composition as that of the target has been obtained under high pressure and strong electron scattering by gas particles. Figure 2 shows how the component of the film formed by the method shown in Figure 1(b) depends on the substrate temperature and gas pressure. The film and target compositions agree within the measurement error, and films with stoichiometric composition are obtained without composition compensation with a wide range of forming conditions. In the case of the $Ba_2YCu_2O_y$ film, no composition discrepancies have been confirmed up to the temperature range approaching this system's melting point, i.e., up to a substrate temperature of 800°C or more. These results show that superior composition controllability has been obtained nearly up to the decomposition temperature which is unique to the substance.

Table 1. Relationship Between Composition and Atmospheric Pressure of the Film Formed by the Method in Figure 1(a)

Gas pressure	Condition (a)
60 m Torr	$Ba_{1.8}Y_{1.0}Cu_{3.0}$
10 m Torr	$Ba_{1.2}Y_{1.0}Cu_{0.8}$

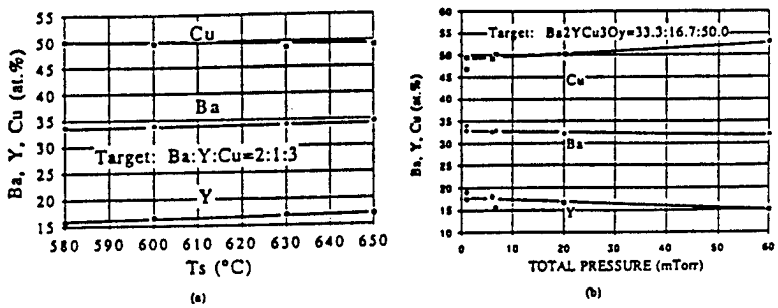

Figure 2. Dependence of Composition of Ba-Y-Cu-O Film Formed by the Method of Figure 1(b) on Substrate Temperature (a) and Atmospheric Pressure (b)

Also, when film formation occurred in the Bi-Sr-Cu-O system, which is a superconductor at the 105 degree level, with a $Bi_2Sr_2Ca_2Cu_3O_y$ stoichiometric composition target under the same conditions, a film of almost the same composition (within 3 at. percent) as that of the target was obtained almost up to the substrate temperature of 800°C.

In both cases, the substrate temperature range in which good composition controllability is obtained far exceeds the crystallization temperature of the superconductive phase, and the crystalline film with the desired composition can be formed with good repeatability.

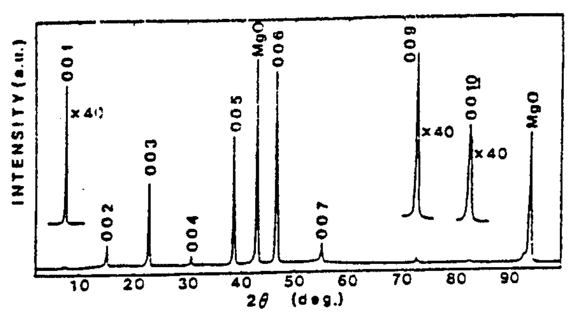

Figure 3(a). X-Ray Diffraction Pattern of the $Ba_2YCu_3O_y$ As-Grown Film Formed by the Method Shown in Figure 1(b)

Figure 3(a) shows the X-ray diffraction pattern of the $Ba_2YCu_3O_y$ film formed by the developed method. Since, with this system, the conduction direction in which the superconductive critical current is high is on the C surface, the C surface should be oriented parallel with the film surface when the system is used as a conductive material. The figure shows strong C surface diffraction lines, indicating that the film is a C-surface-oriented film. Figure 3(b) shows a sample of the temperature dependence of the electrical resistance of such a film. Since not only is an 88 K or higher zero resistance temperature obtained, but also the extrapolation value of conductive resistance to 0 K passes the zero point, it is understood that a high-quality film with few defects is obtained. Also, since these films have a film composition that agrees with stoichiometry and are as-grown, they are superior in surface smoothness, do not show macroscopic organizations up to magnification of

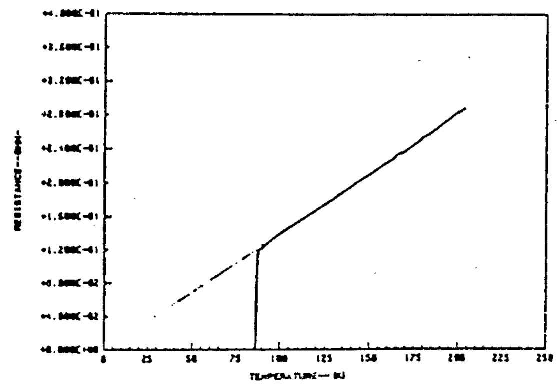

Figure 3(b). Temperature Characteristics of $Ba_2YCu_3O_y$ As-Grown Thin Film Formed by the Method Shown in Figure 1(b)

50,000 times in surface observation with a scanning electron microscope, and show 20 Å or less unevenness when measured with a surface roughness meter.

Table 2 shows the crystalline structure and the relationship between Tc and substrate temperature for the Bi-Sr-Ca-Cu-O film formed by the method shown in Figure 1(b) with the $Bi_2Sr_2Ca_2Cu_4O_y$(2-2-2-4) and $Bi_2Sr_2Ca_2Cu_3O_y$(2-2-2-3) targets. As mentioned earlier, the film composition agreed with that of the target to within 3 percent up to a substrate temperature of 800°C. Crystallization of the superconductive phase starts at about 600°C, the 2-2-1-2 phase with a critical temperature 80 K grows in the range of a substrate temperature of 600~720°C, and the 2-2-2-3 phase at the critical temperature of 100 K level starts mixing when the temperature is 720°C or more. If the 2-2-2-3 target with the latter stoichiometric composition is used, a rising of the zero resistance temperature and enhancement of surface smoothness are observed.

Table 2. Relationship Among Crystalline Structure, Critical Temperature, and Substrate Temperature of the Bi-Sr-Ca-Cu-O Film Formed by the Method Shown in Figure 1(b)

Used target	Substrate temperature		
	<580 °C	600~720°C	>720°C
2-2-2-4	Amorphous	2-2-1-2+CuO	2-2-2-3 +2-2-2-3+CuO
2-2-2-3	Amorphous	2-2-1-2+CuO	2-2-1-2+2-2-2-3
		Tc onset	
2-2-2-4	Semiconductor	70, 80 K	70, 80, 105 K (few)
2-2-2-3	Semiconductor	70, 80 K	80, 105 K

Performance

This thin film synthesis technique has composition controllability to within 3 percent for an element of high vapor pressure, with better composition controllability for an element of low vapor pressure under wide ranges of

atmospheric pressure and substrate temperature, and is effective for enhancing repeatability and artificial composition modulation in sputtering synthesis of high-quality oxide superconductor thin films. Also, since the target composition agrees with film composition, thin films can be made from a single target, even for multielement oxide superconductors, and incorporation into an integrated process is facilitated.

Application Fields

This technique is applicable to general sputtering synthesis of oxide thin films.

Related Patents

Patent applied for "Superconductive thin film manufacturing device"

Patent applied for "Sputtering device for oxide thin film fabrication"

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